



Naval Facilities Engineering Command Southwest  
BRAC PMO West  
San Diego, CA

**FINAL, REVISION 1  
PARCEL G REMOVAL SITE EVALUATION WORK PLAN  
ADDENDUM**

Radiological Investigation, Survey, and Reporting  
Parcel G

FORMER HUNTERS POINT NAVAL SHIPYARD  
SAN FRANCISCO, CALIFORNIA

July 2020

**Approved for public release: distribution unlimited.**

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Prepared for:



Department of the Navy  
Naval Facilities Engineering Command Southwest  
BRAC PMO West  
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Lisa Berdik, PE  
Project Manager

July 24, 2020

Date

## Table of Contents

<b>LIST OF TABLES .....</b>	<b>II</b>
<b>LIST OF APPENDICES .....</b>	<b>II</b>
<b>ACRONYMS AND ABBREVIATIONS .....</b>	<b>III</b>
<b>1.0 INTRODUCTION .....</b>	<b>1-1</b>
1.1 Scope of Work .....	1-1
1.2 Project Schedule .....	1-1
1.3 Project Organization .....	1-1
1.4 Site Safety .....	1-1
1.5 Radiation Protection Program .....	1-2
1.6 Removal Site Evaluation Work Plan Addendum Organization .....	1-2
<b>2.0 PROJECT REQUIREMENTS .....</b>	<b>2-1</b>
2.1 Accident Prevention Plan/Site Safety and Health Plan .....	2-1
2.2 Radiation Protection Plan .....	2-1
2.3 Sampling and Analysis Plan .....	2-1
2.4 Contractor Quality Control Plan .....	2-1
2.5 Environmental Protection Plan .....	2-1
2.5.1 Stormwater Management Plan .....	2-1
2.5.2 Dust Management and Air Monitoring Plan .....	2-1
2.6 Waste Management Plan .....	2-2
2.7 Soil Sorting Plan .....	2-2
<b>3.0 RADIOLOGICAL SURVEY .....</b>	<b>3-1</b>
3.1 Gamma Instruments .....	3-1
3.2 Survey Investigation Levels .....	3-2
3.3 Instrument Detection Calculations .....	3-2
3.3.1 Gamma Surface Activity .....	3-3
3.3.2 Gamma Scan Minimum Detectable Concentration .....	3-3
3.4 Calibration .....	3-6
3.5 Daily Performance Checks .....	3-6
3.6 Debris Screening .....	3-7
3.7 Sampling of Diverted Material .....	3-9
<b>4.0 REFERENCES .....</b>	<b>4-1</b>

## List of Tables

---

Table 1	Project Schedule
Table 2	Key Project Personnel
Table 3	Survey Instruments
Table 4	A Priori Scan Minimum Detection Concentrations—Gamma Instrumentation
Table 5	A Priori Static Minimum Detection Concentrations—Alpha/Beta Instrumentation

## List of Appendices

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Appendix A	Responses to Comments
Appendix B	Sampling and Analysis Plan Addendum (dated January 2020)
Appendix C	Contractor Quality Control Plan
Appendix D	Stormwater Management Plan
Appendix E	Dust Management and Air Monitoring Plan (dated July 2020   DCN: APTM-0006-5065-0005.A1/F.R1)
Appendix F	ISO-PACIFIC Soil Sorting Operations Work Plan
Appendix G	Gamma Scan Minimum Detectable Concentration Calculations

## Acronyms and Abbreviations

$^{137}\text{Cs}$	cesium-137
$^{226}\text{Ra}$	radium-226
$^{232}\text{Th}$	thorium-232
$\mu\text{R/hr}$	microrentgen per hour
ALARA	as low as reasonably achievable
APP/SSHP	<i>Accident Prevention Plan, Radiological Investigation, Survey, and Reporting, Parcel G, Former Hunters Point Naval Shipyard, San Francisco, California</i>
APTIM	Aptim Federal Services, LLC
cm	centimeter
cpm	counts per minute
$\text{g/cm}^3$	gram per cubic cm
HPNS	Former Hunters Point Naval Shipyard
keV	kiloelectron volts
MARSSIM	<i>Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)</i>
MDC	minimum detectable count
MDCR	minimum detectable count rate
NaI	sodium iodide
Navy	U. S. Department of the Navy
NRC	U.S. Nuclear Regulatory Commission
RPP	<i>Radiological Prevention Plan, Radiological Investigation, Survey, and Reporting, Parcel G, Former Hunters Point Naval Shipyard, San Francisco, California.</i>
SAP	Sampling and Analysis Plan
SU	survey unit
WP	<i>Final Parcel G Removal Site Evaluation Work Plan, Former Hunters Point Naval Shipyard, San Francisco, California</i>
WPA	work plan addendum

## **Appendix E**

# **Dust Management and Air Monitoring Plan**

(dated July 2020 | DCN: APTM-0006-5065-0005.A1/F.R1)



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## **APPENDIX E**

### **FINAL, REVISION 1 DUST MANAGEMENT AND AIR MONITORING PLAN**

Radiological Investigation, Survey, and Reporting  
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FORMER HUNTERS POINT NAVAL SHIPYARD  
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# Table of Contents

LIST OF FIGURES .....	II
LIST OF TABLES .....	II
LIST OF ATTACHMENTS .....	II
ACRONYMS AND ABBREVIATIONS .....	II
1.0 INTRODUCTION .....	1-1
2.0 MANAGEMENT PRACTICES FOR CONTROL OF FUGITIVE DUST EMISSIONS .....	2-1
2.1 Potential Sources of Fugitive Dust .....	2-1
2.2 General Construction Dust-Control Methods .....	2-1
2.2.1 Construction Traffic .....	2-2
2.2.1.1 Track-Out Prevention .....	2-2
2.2.1.2 Traffic Control .....	2-2
2.2.2 Site Preparation .....	2-3
2.2.3 Material Stockpiles .....	2-3
2.2.4 Transport of Solid Bulk Material .....	2-3
2.2.5 Excavation Activities .....	2-4
2.2.6 Soil Processing and Management .....	2-4
2.2.7 Backfill Activities .....	2-5
2.2.8 Post-Construction Stabilization of Disturbed Areas .....	2-5
2.2.9 Recycling .....	2-6
2.2.10 Wind Speed and Air Monitoring and Response .....	2-6
3.0 AIR QUALITY MONITORING PROCEDURES .....	3-1
3.1 Air Quality Sampling and Real-Time Dust-Monitoring Locations .....	3-2
3.2 Air Quality Sample Collection and Testing Procedures .....	3-3
3.2.1 Total Suspended Particulates, Manganese, and Lead .....	3-3
3.2.2 PM10 .....	3-4
3.2.3 Asbestos .....	3-4
3.2.4 Real-Time Dust Monitoring .....	3-5
3.2.4.1 Personnel Dust Monitoring .....	3-5
3.2.4.2 Dust Monitoring for Off-site Receptors .....	3-6
3.2.5 Radionuclides of Concern .....	3-7
3.3 Field Quality Control Procedures .....	3-9
3.3.1 Calibration Flow Rates and Maintenance .....	3-9
3.3.2 Sample Documentation, Custody and Field Logs .....	3-10
4.0 DATA REVIEW AND REPORTING .....	4-1
5.0 REFERENCES .....	5-1

## List of Figures

---

Figure 1	Parcel G Air Sampling and Dust Monitoring Locations
Figure 2	Wind Rose, Parcel E
Figure 3	Wind Rose, Parcels B-1, C, and UC-2

## List of Tables

---

Table 1	Dust-Monitoring and Air-Sampling Threshold Criteria
Table 2	Radionuclides of Concern Airborne Concentration Action Levels
Table 3	Air-Sampling and Dust-Monitoring Frequency and Sample Collection Methods
Table 4	Field and Laboratory Quality Control Samples
Table 5	Air-Sampling Unit Flow Checks and Controls

## List of Attachments

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Attachment 1	Crosswalk
Attachment 2	Field Forms
Attachment 3	Work Instruction and Standard Operating Procedures

## Acronyms and Abbreviations

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µg/m <sup>3</sup>	microgram per cubic meter
APTIM	Aptim Federal Services, LLC
BAAQMD	Bay Area Air Quality Management District
CFR	Code of Federal Regulations
CSAAQS	California State ambient air quality standard
DAC	derived airborne concentration
DMP	Dust Management and Air Monitoring Plan
DTSC	California Department of Toxic Substances Control
EPA	U.S. Environmental Protection Agency
mph	mile per hour
Navy	U.S. Department of the Navy
PDR	personal data ram
PM10	particulate matter smaller than 10 microns in diameter
QC	quality control
ROC	radionuclide of concern
SOP	standard operating procedure
TSP	total suspended particulate

## 1.0 INTRODUCTION

This Dust Management and Air Monitoring Plan (DMP) identifies procedures to reduce fugitive dust emissions (dust management practices), and for the air quality monitoring of fugitive dust emissions, that may be generated during the radiological removal activities at Parcel G, Former Hunters Point Naval Shipyard, San Francisco, California (Figure 1). At the request of the U.S. Environmental Protection Agency (EPA), the U.S. Department of the Navy (Navy) has included additional dust monitoring and air sampling procedures in this DMP in order to proceed with the associated fieldwork.

Fugitive dust emissions will be minimized and controlled during work hours, evenings, weekends, and holidays. Inactive surface areas and storage piles (defined as inactive for more than seven calendar days) will be stabilized with a chemical stabilizer. Active surface areas and storage piles (areas where material is being added or removed within seven calendar days) will be wetted with water and/or a chemical stabilizer as appropriate. At a minimum, water will be used for dust control during the week, and chemical stabilizer or equivalent will be applied to active stockpiles at the end of each workday or as needed during high winds. The dust management portion of the DMP describes measures to address the substantive requirements of the Bay Area Air Quality Management District (BAAQMD) (2015) *Particulate Matter and Visible Emissions*.

The air quality monitoring section of the DMP will ensure on-site worker safety and provide reasonable assurance of the protection of the surrounding residents and public receptors from fugitive dust emissions that may be generated during the radiological removal activities at Parcel G. The nearest residential receptors are located at The San Francisco Shipyard at 11 Innes Court, approximately 170 meters north of the Parcel G boundary. The nearest public receptors are at Building 606 located approximately 50 meters south of the Parcel G boundary. In addition, approximately 110 meters north of Parcel G, public receptors are present at a commercial kitchen and artist studios in the 100 block of Horne Avenue. The air quality monitoring is appropriate to assess potential impacts to the nearby residents and public receptors, in addition to on-site workers. The southwest monitoring station will assess potential microscale impacts to public receptors at Building 606. The northwest and northeast monitors will assess potential middle scale impacts to residents and public receptors within 500 meters of the site. Three types of air monitoring will be conducted during intrusive construction activities, and while stockpiles are present and workers are on site, and include the following:

- Air quality sampling for total suspended particulates [TSP], lead, manganese, particulate matter smaller than 10 microns in diameter [PM10], and asbestos at the Parcel G perimeter
- Real-time dust monitoring at the Parcel G perimeter and at daily work areas
- Radionuclides of concern (ROCs) air monitoring at the Parcel G perimeter and at daily work areas

A project sign will be installed near the site entrance or other appropriate location. The sign will include project contact information for both the Navy and Aptim Federal Services, LLC (APTIM) personnel for reporting of dust or other air quality concerns.

## 2.0 MANAGEMENT PRACTICES FOR CONTROL OF FUGITIVE DUST EMISSIONS

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The following subsections describe engineering controls of fugitive dust at the site.

### 2.1 Potential Sources of Fugitive Dust

Planned site activities have the potential to generate air emissions in the form of fugitive dust. Possible sources of emissions include the following activities:

- Construction Traffic—Moving construction equipment around the construction areas is capable of creating construction emissions in work areas.
- Site Preparation—Removing vegetation will increase the potential for fugitive dust emissions through wind erosion.
- Excavation—Removing soil from the ground and loading it either onto screening pads or into waiting vehicles could cause fugitive dust emissions.
- Transportation of Solid Bulk Material—Transporting excavated soil from excavations for radiological screening and/or disposal. Material will be reused on site to the extent practicable for backfill. If soil is left uncovered, fugitive emissions could occur.
- Material Stockpiles—Soil that has been radiologically processed will be stockpiled prior to being used as backfill. Soil will be loaded into trucks to relocate piles on site as necessary. Fugitive emissions during stockpiling and truck loading, as well as wind erosion, are possible.
- Subgrade Preparation—Cutting and placing backfill soil could cause fugitive dust emissions.
- Site Restoration—Backfilling and restoration of the excavated areas may produce fugitive dust emissions.
- Fugitive dust emissions generated upgradient of the site (off site) may also constitute a source.

### 2.2 General Construction Dust-Control Methods

Control methods for fugitive dust are described for the following emissions generated from the construction activities at the project site:

- Dust entrained during on-site travel on paved and unpaved surfaces
- Dust entrained during surface cover, excavation, material screening, backfill, and final grading at the construction site
- Dust entrained during material stockpiling, and loading and unloading operations

- Wind erosion of areas disturbed during removal activities
- Vehicle emissions associated with construction equipment
- Water applied as needed to control visible dust

The Construction Manager is ultimately responsible to identify and control visible dust; however, site personnel will be trained to recognize visible dust and report it immediately to site management. Observations will be made throughout construction, especially during earth moving activities.

## 2.2.1 Construction Traffic

This subsection describes dust-control methods for construction traffic.

### 2.2.1.1 Track-Out Prevention

Track-out of loose materials will be controlled by use of tire-cleaning rumble grid plates at the access point from the project site to the paved road to prevent track-out of mud or loose soil onto roadways. These track-out prevention-control points will be established at the primary site access point. To ensure that tires are free of mud or loose soil prior to leaving the site egress, bulk-loaded trucks and commercial vehicles will be required to pass over a gravel pad (50 feet in length) and over the rumble grid plates where the soil residue from the tires will be removed. The accumulated soil will be routinely removed and managed as waste. Visible track-out onto a paved road where vehicles exit the work site will be removed by wet sweeping.

### 2.2.1.2 Traffic Control

The following mitigation measures will be followed for fugitive dust emissions from construction traffic traveling on paved streets:

- Bulk-loaded trucks used for transportation of soil and other heavy earth-moving equipment will not be allowed to exit the construction sites, except through the track-out prevention-control point.
- Construction areas adjacent to and above grade from a paved roadway will be treated with best management practices, as specified in the Stormwater Management Plan (Appendix C of this Work Plan Addendum).
- Roadways within the site will be swept using a wet sweeper or washed down to remove soil. The accumulated soil will be routinely removed from non-traffic areas such as gutters and curbs and managed in accordance with the waste management plan section of the *Final Parcel G Removal Site Evaluation Work Plan, Former Hunters Point Naval Shipyard, San Francisco, California* (CH2M Hill, Inc., 2019).
- No vehicle will exceed 15 miles per hour (mph) within the construction site and 5 mph in work areas.

If the preceding mitigation methods fail to properly control fugitive dust emissions, one or more of the following reasonably available control measures will be applied:

- Unpaved active portions of the construction sites will be watered or treated with dust-control solutions to minimize windblown dust and dust generated by vehicle traffic.
- Paved portions of the construction sites (including traffic routes) will be cleaned more frequently to control windblown dust and dust generated by vehicle traffic.
- Vehicle trips will be reduced if necessary.

## 2.2.2 Site Preparation

Fugitive dust emissions from site preparation, including clearing and grubbing, will be controlled using the following methods:

- Following clearing and grubbing, exposed surfaces will be wetted to sufficiently maintain soil-moisture content to minimize fugitive dust creation.
- Unpaved, inactive portions of the work area under construction will be watered, or a chemical soil stabilizer will be applied to minimize fugitive dust creation.

## 2.2.3 Material Stockpiles

Fugitive dust emissions from soil storage piles will be controlled by using a temporary cover, water, or a chemical soil stabilizer.

## 2.2.4 Transport of Solid Bulk Material

Fugitive dust emissions from trucks used to transport excavated and backfill material will be controlled using the following methods:

- Trucks that are used to transport solid bulk material will be covered (tarp) prior to leaving the site.
- Vehicles will be checked to ensure that they are tarped and to remove excess material on the shelf or exterior surfaces of the cargo compartment.
- Bulk-loaded trucks will exit the work site via an established track-out control point.
- Truck tires will be inspected and dry brushed to remove dirt prior to leaving exclusion zones. Wet sweeping will be implemented as needed if track out of dirt is observed. Accumulated soil will be managed in accordance with the waste management plan section of the *Final Parcel G Removal Site Evaluation Work Plan, Former Hunters Point Naval Shipyard, San Francisco, California* (CH2M Hill, Inc., 2019).

## 2.2.5 Excavation Activities

Fugitive dust emissions from excavation and loading activities will be controlled using the following methods:

- Soil will be wetted prior to excavation activities to reduce dust migration. Additional water will be added during active excavation, material handling, and loading on an as-needed basis. Active excavation areas will be wetted approximately every two hours or more frequently if needed, during periods of dry weather and/or windy conditions. A water truck or water buffalo will be dedicated to excavation and removal operations.
- The height from which excavated soil is dropped to trucks, stockpiles, or screening pads will be minimized.
- Trucks will be equipped with tarping systems to cover loads during soil transport.
- Truck traffic will be minimized to the shortest allowable haul routes from the work areas, screening area, and stockpile areas.
- Chemical soil stabilizer or straw mulch will be applied in sufficient quantities to disturbed areas to create a stabilized surface.
- Backfill materials will be wetted on an as-needed basis to maintain moisture. Loader buckets will be emptied slowly and drop heights from loader buckets will be minimized. A water truck or water buffalo will be dedicated to backfilling operations.
- Water, a temporary cover, or chemical soil stabilizer will be applied to control fugitive dust emissions from stockpiled material when not actively handled.

## 2.2.6 Soil Processing and Management

Fugitive dust emissions from radiologically processing of soil through the soil sorter will be controlled using the following methods:

- Soil will be wetted prior to soil-moving activities to reduce dust migration. Additional water will be added during soil processing on the conveyor, material handling, and loading on an as-needed basis. Active work areas will be wetted approximately every two hours or more frequently if needed, during periods of dry weather and/or windy conditions. A water truck or water buffalo will be dedicated to soil processing operations.
- The height from which soil is dropped onto the ground, trucks, stockpiles, or conveyor equipment will be minimized.
- Materials will be wetted on an as-needed basis to maintain moisture. Loader buckets will be emptied slowly and drop heights from loader buckets will be minimized. A water truck or water buffalo will be dedicated to backfilling operations.
- Trucks will be equipped with tarping systems to cover loads during soil transport.



- Truck traffic will be minimized to the shortest allowable haul routes from the work areas and stockpile areas.
- Chemical soil stabilizer or straw mulch will be applied in sufficient quantities to disturbed areas so as to create a stabilized surface.
- Water, a temporary cover, or chemical soil stabilizer will be applied to control fugitive dust emissions from stockpiled material when not actively handled, at the end of each workday for active stockpiles, or as needed during high winds.

### **2.2.7 Backfill Activities**

Fugitive dust emissions from backfill activities (cutting, filling, and grading material) will be controlled using the following methods:

- Soil will be wetted prior to soil-moving activities to reduce dust migration. Additional water will be added during active cutting (excavation), material handling, and loading on an as-needed basis. Active work areas will be wetted approximately every two hours or more frequently if needed, during periods of dry weather and/or windy conditions. A water truck or water buffalo will be dedicated to earthmoving operations.
- The height from which soil is dropped onto the ground, trucks, or stockpiles will be minimized.
- Trucks will be equipped with tarping systems to cover loads during soil transport.
- Truck traffic will be minimized to the shortest allowable haul routes from the work areas and stockpile areas.
- Chemical soil stabilizer or straw mulch will be applied in sufficient quantities to disturbed areas so as to create a stabilized surface.
- Backfill materials will be wetted on an as-needed basis to maintain moisture. Loader buckets will be emptied slowly and drop heights from loader buckets will be minimized. A water truck or water buffalo will be dedicated to backfilling operations.
- Water, a temporary cover, or chemical soil stabilizer will be applied to control fugitive dust emissions from stockpiled material when not actively handled.

### **2.2.8 Post-Construction Stabilization of Disturbed Areas**

Disturbed areas will be restored to existing site conditions, which includes re-establishing concrete swales and asphalt roadways. Therefore, fugitive dust from remaining open areas will not be an issue.

## 2.2.9 Recycling

Non-impacted asphalt and concrete are typically recycled on site and may produce fugitive dust emissions. Fugitive dust emissions from recycling activities will be controlled using the following methods:

- Asphalt and concrete will be wetted prior to handling to reduce dust migration. A water truck or water buffalo will be dedicated to this activity.
- Additional water will be added during active grinding, sorting, material handling, and loading, as needed, to control fugitive dust.
- The height from which crushed material is dropped to trucks, stockpiles, or pads will be minimized.
- Trucks will be equipped with tarping systems to cover loads during transport.
- Truck traffic will be minimized to the shortest haul routes from the work areas and stockpile areas.
- A chemical soil stabilizer will be applied in sufficient quantities to stockpiles to create a stabilized surface.

## 2.2.10 Wind Speed and Air Monitoring and Response

Because wind speed and direction have a direct influence on the potential for dust generation and downwind off-site dispersion, wind speed and direction will be monitored throughout the day via an on-site meteorological station. The on-site meteorological station will be located at the Former Hunters Point Naval Shipyard site trailer in Parcel C (Figure 1). The on-site meteorological station data will be periodically verified with off-site weather station data. The predominant wind direction at Former Hunters Point Naval Shipyard is from the west or west-northwest. Wind roses from adjacent Parcel E and Parcels B-1, C, and UC-2 are provided on Figures 2 and 3, respectively.

As a general guide, dust-control measures will be evaluated and implemented in response to the potential for specific wind speed conditions that may exacerbate dust generation. Wind speed will be monitored by the on-site meteorological station throughout the day while construction activity is occurring. Wind speeds will be continuously monitored and recorded in 10-minute, time-weighted intervals. The exceedance of a threshold wind speed is defined as the point at which the threshold wind speed is exceeded during two consecutive 10-minute time-weighted intervals.

During earthmoving activities, a response action will be taken when the wind speed exceeds a threshold level for two consecutive 10-minute, time-weighted intervals, or if real-time air monitoring data has sustained exceedances over a 15-minute interval. In addition, a windsock, designed to fully respond to a 15-mph wind speed will be installed at the site (Figure 1). The windsock will be monitored during earthmoving activities and will serve as a qualitative indicator of real-time wind speed and direction.

Response actions will be taken on the basis of ranges of wind speed conditions or monitoring data. When the recorded wind speed is between 0 and 15 mph, standard best management dust-control practices will be implemented (Sections 2.2.1 through 2.2.7). When the recorded wind speed is between 15 and 20 mph for two consecutive 10-minute time-weighted intervals, the construction foreman will conduct a sitewide survey on 60-minute intervals for evidence of wind-generated dust. In the event that wind-generated dust is observed or measured above the threshold, the foreman will increase the frequency of watering unpaved haul roads, wetting and/or stabilizing disturbed soil and street sweeping as needed. Truck traffic may also be reduced.

When the recorded wind speed is between 20 and 25 mph for two consecutive 10-minute, time-weighted intervals, the construction foreman will immediately increase the frequency of watering unpaved haul roads, wetting and/or stabilizing disturbed soil, and street sweeping as needed. Truck traffic may also be reduced. The foreman will conduct a sitewide survey on 30-minute intervals for evidence of wind-generated dust.

In the event that wind-generated dust is observed or measured despite the application of the increased dust-control measures, the foreman will discontinue the work producing the dust until the wind speed is recorded below 20 mph for two consecutive 10-minute, time-weighted intervals and measured real-time dust (PM<sub>10</sub>) concentrations have returned to background or less than 40 µg/m<sup>3</sup>.

When the recorded wind speed exceeds 25 mph for two consecutive 10-minute, time-weighted intervals, dust-generating work will stop until the wind speed drops below 25 mph for two consecutive 10-minute, time-weighted intervals.

The meteorological station will be programmed to send out a notification alarm to the construction foreman when the wind speed has reached wind speeds of 15, 20, and 25 mph for two consecutive, 10-minute time-weighted intervals.

## 3.0 AIR QUALITY MONITORING PROCEDURES

Air quality monitoring of fugitive dust emissions that may be generated during the radiological removal activities at Parcel G will be performed to ensure on-site worker safety and provide reasonable assurance of the protection of the surrounding residents and public receptors. Air quality monitoring equipment is operated and maintained under the direction and oversight of the APTIM Project Chemist and the APTIM Project Radiation Safety Officer. Any personnel involved in sampling air quality will be trained by Project Chemist or Project Radiation Safety Officer prior to any field activities. The training will be recorded in the field activity daily logs for the project.

Three types of air monitoring will be conducted during intrusive construction activities, and while stockpiles are present and workers are on site, and include the following:

- Air quality sampling for TSPs, lead, manganese, PM<sub>10</sub>, and asbestos at the Parcel G perimeter
- Real-time dust monitoring at the Parcel G perimeter and at daily work areas
- ROCs air monitoring at the Parcel G perimeter and at daily work areas

Analytical results will be compared to the air-sampling threshold criteria listed in Tables 1 and 2. If project activities are the cause of exceedances at real-time monitoring locations, additional control measures, as described in Section 2.2, will be implemented immediately. The Navy will be notified of exceedances and additional control measures within 24 hours. The Navy will notify regulatory agencies. If laboratory data from the filter-based air samplers exceeds the threshold criteria, the Navy will be notified. The data will be compared to the real-time dust monitoring, wind speed data, and Daily Contractor Production Reports, to determine the cause of any exceedances. Additional control measures will be implemented immediately, and an evaluation of the control and measurement strategy will be implemented. The Navy will notify regulatory agencies and share all available information regarding the exceedance. The Navy will consider requesting a quick-turn analysis of the next batch of filters to more quickly determine if exceedances are ongoing. If laboratory data shows exceedances that the continuous data do not, the Navy will evaluate the appropriateness of monitoring locations, sampling methods, and sampling frequency.

BAAQMD implements the California State ambient air quality standard (CSAAQS) for the Bay Area. The CSAAQS is designed to protect the general public from airborne particulates generated in the urban, suburban, and rural environments. The CSAAQS is not meant to be applied to general project-specific construction actions and related air quality. Rather, the standard is used to attain city- or regional-wide ambient air quality goals for the benefit of the general public. The current CSAAQS for PM<sub>10</sub> is 50 micrograms per cubic meter ( $\mu\text{g}/\text{m}^3$ ) average per 24-hour day. The City and County of San Francisco is currently a non-attainment area for the CSAAQS for PM<sub>10</sub>. Dust control measures will be

implemented if real-time air monitoring data has sustained exceedances of  $50 \mu\text{g}/\text{m}^3$  over a 15-minute interval, as described in Section 2.2. The upwind and downwind dust monitors will enable emissions from off site to be considered in the  $50 \mu\text{g}/\text{m}^3$  average per 24-hour day action level comparison, when wind speeds are greater than 5 mph and wind direction is constant over the sampling period. There will be situations, like stagnant conditions or when the wind direction varies during the data collection interval, where consideration of upwind is not appropriate. Reporting will include wind data and footnotes describing how and when upwind and downwind monitors have been used to exclude off-site emissions in air quality monitoring reports.

### 3.1 Air Quality Sampling and Real-Time Dust-Monitoring Locations

Air quality sampling and real-time dust monitoring are performed to estimate and assess the impact of the field activities to on-site receptors (i.e., workers) and off-site receptors (i.e., the public). Representative meteorological data specifically wind speed and direction, will be used to identify the most appropriate locations for the air-monitoring stations. The locations may be modified as needed for accessibility considerations and worker safety. Air quality sampling and real-time dust-monitoring stations will remain stationary while sampling is conducted.

Air quality sampling stations will be used to measure TSP, metals, PM<sub>10</sub>, asbestos, and ROCs. Real-time dust monitoring will be used to monitor employee work areas and at locations near the perimeter of the site to represent potential exposure for members of the public working or residing adjacent to Parcel G.

Figure 1 shows the proposed locations for air quality samples and real-time dust-monitoring stations. The air sampling station locations are based on the locations established in the *Final Basewide Dust Control Plan, Revision 1, Hunters Point Shipyard, San Francisco, California* (Tetra Tech EC, Inc, 2010), which provides consistency across multiple projects and parcels. The actual air sampling and dust monitoring locations will be sited to represent ambient air and will be a sufficient distance from physical obstructions, non-site sources, and site sources to the extent practicable to obtain representative data. air flow around buildings and obstruction will also be considered when establishing monitoring locations. There will be a minimum of two air-sampling stations, upwind Station 1 and downwind Station 17 or 17A, and three real-time dust monitor stations, placed at the northwest, northeast, and southwest corners of Parcel G (Figure 1). For air sampling stations with high-volume particulate samplers, sampler inlets will be placed approximately 2 meters away from high-volume samplers. Inlets for collocated real-time dust monitors will be placed one meter apart. Inlet heights for air samplers and the dust monitors range from approximately 4.5 to 5.5 feet above the ground surface. Weather forecasts will be checked daily at [www.noaa.gov](http://www.noaa.gov) to determine the prevailing wind direction(s).

Personnel real-time dust monitors, used for worker health and safety, are not show on Figure 1 as they are located within the footprint the daily work areas and move accordingly.

Radiological air monitoring will be conducted at upwind Station 1 and downwind Station 17 or 17A, shown on Figure 1, in accordance with the applicable radiation work permit requirements and *APTIM Management System* (AMS)-710-07-PR-04011, “Radiological Surveys and Monitoring” (APTIM, 2020). Additional radiological air monitors may be placed within the daily work areas to monitor for worker health and safety. These additional radiological air monitors are not shown on Figure 1 as they are relocated often based on daily work activities.

## 3.2 Air Quality Sample Collection and Testing Procedures

The air quality sampling for this project include portable air quality monitoring stations and real-time dust monitors that will be used to perform monitoring during field activities. Air quality sampling equipment is setup approximately 15 to 30 minutes before fieldwork is scheduled to begin. No earthmoving work will start until the sampling equipment is running. Air monitoring, as described in Section 1.0, will continue while stockpiles remain and workers are on site at Parcel G. The workday is estimated to range from a minimum of 4 hours to maximum of 14 hours per day. Samples will be collected at the air quality stations and will be analyzed for the airborne contaminants of concern, including TSP, lead, and manganese, PM<sub>10</sub>, asbestos, and ROCs. Filter-based particulate samplers will monitor for 24,  $\pm 1$ , hours to represent each workday and the following night to collect a sample that is consistent in duration to the PM<sub>10</sub> standard. The air-sampling stations will be supplemented with real-time dust monitoring. The air quality sampling will be used to assess the status of air quality compliance and to evaluate modifications to project activities in the event of compliance concerns.

Filter-based samplers for TSP (including metals), PM<sub>10</sub>, and asbestos will monitor for 24,  $\pm 1$ , hours to represent each workday and the following night. Filter-based samplers for ROCs will monitor for 104 hours to represent the workweek. If there have been no sustained exceedances of 50  $\mu\text{g}/\text{m}^3$  over a 15-minute interval from the real-time dust monitors on a Friday (or day preceding a holiday), samples may be collected at the end of that workday.

Tables 1 and 2 show the fugitive dust contaminants of concern for the site, regulatory limits and selected methodology sensitivity (reporting limit) to ensure the selected monitoring methods will achieve the project action limits. Table 3 lists sampling frequency and sampling methods for each air quality and real-time dust-monitoring station. Table 4 provides the field and laboratory quality control samples and laboratory standard operating procedure (SOP) references, and Table 5 provides the air-sampling equipment unit flow checks, controls, and maintenance.

### 3.2.1 Total Suspended Particulates, Manganese, and Lead

Air quality sampling TSP will be sampled with a high-volume (39 to 60 cubic feet per minute) air sampler (HI-Q or Tisch high-volume sampler, or equivalent) in accordance with EPA’s reference sampling method for TSP, described in Title 40 Code of Federal Regulations (CFR), Part 50, Appendix B. Each sample will be collected on a laboratory prepared, pre-weighed filter over the course of a period not to exceed 25 hours; the filter will then be returned to the laboratory and weighed to determine the amount of TSP

collected. Once the filter weight has been determined, the sample will be further analyzed for manganese and lead in accordance with one of the IO-3 methods identified in EPA's *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air* (1999). The equipment specifications and sampling procedures will comply with the specifications provided in the regulations. The general procedures for sample collection are as follows:

1. Obtain pre-weighed prepared filters from laboratory.
2. Inspect/clean the unit for accumulated dirt/dust or insects.
3. Put on clean disposable sample gloves and open the filter cassette. Inspect the filter for pinholes, tears, abrasions, loose material, discoloration, and other non-uniformity. Discard any defective or damaged filters. Place new filter (ID # down) on the filter cassette. Record the filter ID number on the sample collection log.
4. Verify enough power supply/fuel for high-volume sampler for full day operation.
5. Turn on motor and allow the flow controller to stabilize (approximately five minutes).
6. Verify flow rate (39 to 60 cubic feet per minute), record starting flow rate and time on sample collection log.
7. End of day, put on a new clean pair of disposable gloves, stop the sampler and record time on sample collection log.
8. Carefully remove filter (care must be taken to not lose any particulate matter from the filter). Fold the filter inward in half (take care not to tear corners). Verify that filter ID matches ID on envelope and what is recorded on sample collection logs. Place inside filter envelope.
9. Label and prepare sample for shipment to the laboratory.

Attachment 3 provides APTIM's high-volume sampling procedure.

### 3.2.2 PM10

PM10 will be sampled with a high-volume (39 to 60 cubic feet per minute) air sampler (HI-Q or Tisch high-volume sampler, or equivalent) in accordance with EPA's reference sampling method for PM10, described in 40 CFR 50, Appendix J. Each sample will be collected on a laboratory prepared, pre-weighed filter over the course of a period not to exceed 25 hours. The filter will be returned to the laboratory and weighed to determine the amount of PM10 collected. Field PM10 sample collection procedures follow the same steps described above for TSP (Section 3.2.1)

### 3.2.3 Asbestos

Asbestos will be sampled and analyzed in accordance with National Institute for Occupational Safety and Health Method 7400, from the *NIOSH Manual of Analytical Methods* (National Institute for Occupational Safety and Health, 1994). Asbestos will be sampled with a low-volume air sampler (Gillian GilAir®

air-sampling pump, or equivalent). National Institute for Occupational Safety and Health Method 7400 requires that samples be collected on three-piece cellulose ester filters fitted with conductive cowlings at a sampling rate of between 0.5 liters per minute and 16 liters per minute. Each sample will be collected over a period not to exceed 25 hours. The general procedures for sample collection are as follows:

1. Obtain 25-millimeter mixed cellulose ester filters from laboratory.
2. Don clean, disposable sample gloves and connect the suction side of the filter to the pump inlet.
3. Place the cassette in the breathing zone height (between 4 to 6 feet from the ground) with the face pointed down (so the face of the cassette points to the ground).
4. Verify enough power supply/fuel for sample pump for full day operation.
5. Turn on motor and allow the flow controller to stabilize (approximately five minutes). For a 25-millimeter cassette, the method recommends a minimum of 400 liters of air at between 0.5 to 16 liters per minute. Field flow rates are set between 2 to 4 liters per minute. Record starting flow rate and time on sample collection log.

NOTE: The asbestos phased contrast microscopy method analysis is adversely affected by accumulations of dust on the filter. The filter will be inspected every two to three hours during the workday. If buildup of dust is observed on the filter, the cassette will be changed, and the times recorded. Both filters will be submitted for analysis.

6. End of day, put on a new, clean pair of disposable gloves, stop the sampler, and record time on sample collection log.
7. Label and prepare sample for shipment to the laboratory. One filter field blank is submitted to the laboratory for every day of sample collection.

Attachment 3 provides APTIM's asbestos sampling procedure.

### **3.2.4 Real-Time Dust Monitoring**

Real-time dust monitoring includes two different procedures and action limits specific to the receptor.

#### **3.2.4.1 Personnel Dust Monitoring**

Data logging real-time aerosol monitors (miniRAM or equivalent) or personal data RAMs (personal data ram [PDR]) will supplement particulate matter 10 microns in diameter readings to provide immediate information for dust levels present in the working zone. The data collected will be used to evaluate the effectiveness of dust-control procedures (in real time) and provide data during the lag time between compound-specific sampling events and laboratory analysis of the samples. The Site Safety and Health Officer will conduct monitoring to ensure that each site worker is adequately protected. In consultation with the Health, Safety, and Environmental Manager, the Site Safety and Health Officer will determine if



personal or additional perimeter monitoring is required to evaluate the potential for personnel exposure.

The personnel monitor will be strategically placed downwind of operations and positioned at an approximate height of 5 feet. PDRs will be placed adjacent to earthmoving activities. They will be checked frequently during the workday and stored data will be downloaded at the end of the work shift. The monitors will be calibrated and operated in accordance with manufacturer's specifications. APTIM will pay special attention to the possibility of high readings generated by water vapor, insects, or equivalent, and thereby the chance of generating false positives. Such observations will be logged. Dust concentrations at or above 1.0 milligram per cubic meter require additional dust suppression be applied for worker health and safety. The personnel health and safety dust monitors are not further discussed in the DMP. For more details about the procedures and documentation forms are found in Appendices A, E, and F of the *Final Accident Prevention Plan, Radiological Investigation, Survey, and Reporting, Parcel G, Former Hunters Point Naval Shipyard, San Francisco, California* (APTIM, 2019).

### 3.2.4.2 Dust Monitoring for Off-site Receptors

In addition, to the high-volume air quality sampling for PM<sub>10</sub> discussed in Section 3.2.2, real-time dust monitoring for PM<sub>10</sub>, using a DustTrak™ II or equivalent, will be performed at site boundaries nearest to publicly occupied areas. Figure 1 shows the dust-monitoring locations specified by California Department of Toxic Substances Control (DTSC) (May 2019). In accordance with the DTSC memorandum (*Human and Ecological Risk Office [HERO] Memorandum, Draft Dust Action Levels for Parcel G, Hunters Point Naval Shipyard, San Francisco, California* [DTSC, 2019]), this additional real-time dust-monitoring data will be compared to the DTSC Human and Ecological Risk Office calculated dust action level of 50 µg/m<sup>3</sup> average per 24-hour day for total PM<sub>10</sub>, which is based on the BAAQMD regulatory limit. Prior to beginning construction activities, background real-time PM<sub>10</sub> data may be collected to establish baseline concentrations. During earthmoving activities, real-time dust levels (PM<sub>10</sub>) will be monitored during the workday to ensure that the dust action levels are not exceeded over a sustained interval of more than 15 minutes, and/or additional dust suppression methods are used if the dust action levels are exceeded. If project activities are the cause of exceedances, additional dust-control measures will be implemented. Section 2.2 describes dust suppression methods. The real-time PM<sub>10</sub> dust-monitoring data will be summarized and provided to the Navy on a weekly basis. The general procedures for DustTrak™ II operation are as follows:

1. Obtain desktop Dust Track™ aerosol monitor (or equivalent monitor) equipped with data logging functions and cloud-based remote telemetry capabilities. This allows for wireless setup, monitoring, and programing directly from a computer.
2. Install the monitor in the environmental protection and tamper-proof security enclosure, which protects the instrument within a waterproof, lockable case.
3. The monitor inlet will be set at an approximate height of 4.5 to 5.5 feet from the ground.

4. Verify there is enough power supply/fuel for sample pump for full day operation.
5. Automatic flow rate is set at 3 liters per minute with a  $\pm 5$  percent tolerance limit. The instrument self-zeroes twice per day.
6. Each instrument will be verified for flow when installed and weekly with a certified flow standard. This is consistent with the instrument manual which states: "There is an internal  $\Delta P$  flowmeter in the DustTrak II instrument that controls flow rate to  $\pm 5$  percent of the factory setpoint. TSI Incorporated recommends checking the flow with an external flow reference meter, especially when collecting data."
7. The DustTrack™ has built-in instantaneous alarm settings with visual, audible, and cloud-based warnings for exceedances, flow rate control, zero control, and filter condition that may indicate maintenance is required. If needed, maintenance or unit replacement is conducted by the APTIM Project Chemist or designee through support from equipment supplier (EquipCo, Concord, California).
8. One instrument will be used for collocated measurements at the southwest monitoring station. This instrument will also be available as a backup instrument as needed. Weekly the samplers will be rotated to different sampling locations such that each sampler is collocated at least one week per month. The criteria for evaluation of dust precision between real-time instruments will be  $\pm 30$  percent over the sampling day.
9. Data logging is continuous 24 hours/7 days a week; however, APTIM downloads data at the end of the workday for reporting purposes.

### 3.2.5 Radionuclides of Concern

Airborne radioactivity monitoring (continuous or grab samples) will be conducted during the course of work. Table 2 shows the ROCs and its respective action levels for workers and the public (off-site receptor).

To control occupational exposures, establish personal protective equipment, and determine respiratory protection requirements, monitoring and trending for airborne radioactive material will be performed as necessary. Each ROC, as specified in Title 10 CFR, Part 20, Appendix B, has a derived airborne concentration (DAC) value. DAC is defined as the concentration in air that will result in an intake of one annual limit if breathed for a working year under light working conditions (inhalation rate of 1.2 cubic meters of air per hour). The annual limit is the derived limit for the quantity of radioactive material intake into the body of a worker by inhalation or ingestion in a year. Engineered controls will be implemented, as described in Section 2.0 if required to maintain airborne concentrations below 10 percent of the applicable DAC value for the ROCs at the sites. Samples collected for worker health and safety will be collected and analyzed following the procedures described in APTIM Work Instruction D2006-4550-011, "Operation and Use of Portable Instruments at Hunters Point Naval Shipyard" (APTIM, 2020) (Attachment 3). Following sample collection, air filters will be allowed to decay for 72 hours

before counting to prevent short-lived radon daughters from interfering with analysis. Following decay, the sample filters will be counted on site using a Ludlum Model 3030 (or equivalent). Air filters indicating positive activity above action levels on initial assessment will be decayed an additional minimum of 72 hours before final on-site assay. If activity above the action level is still indicated after the decay period, the filter will be sent to the off-site laboratory for additional analysis including low background gross alpha and beta analysis and/or isotopic analysis.

Perimeter samples for ROCs will be collected to demonstrate dust management controls are protective of public health for off-site receptors. The ROC sampling locations will be co-located with the air quality sampling locations, upwind Station 1 and downwind Station 17 or 17A, shown on Figure 1. Radiological air samples will be collected using low-volume air samplers, F&J Specialty Products, Inc. Model LV-1D (or equivalent). One sample will be collected over 104 hours (Monday morning to Friday afternoon) to achieve the sample volume required to provide minimum detectable activities below the action levels. A minimum sample volume of 8,640 cubic feet is required for analysis. The samples will be collected following the procedures described in APTIM Work Instruction D2006-4550-011, "Operation and Use of Portable Instruments at Hunters Point Naval Shipyard" (APTIM, 2020) (Attachment 3).

Perimeter samples for ROCs will be analyzed at the off-site laboratory for the following analyses:

- Cesium-137 by gamma spectroscopy according to EPA Method 901.1MOD/DOE EML HASL 300 Method GA-01-R
- Strontium by EPA Method 905.0 or equivalent methods
- Isotopic plutonium by alpha spectroscopy
- Isotopic uranium by alpha spectroscopy
- Isotopic thorium by alpha spectroscopy
- Alpha-emitting radium isotopes by EPA Method 903.0 or equivalent methods

Laboratory SOPs for radionuclide analyses are provided in Attachment 2 to the Sampling and Analysis Plan Addendum (Appendix B of this Work Plan Addendum). Attachment 3 includes the filter preparation SOP (SOP ST-RC-0004) for ROCs.

### 3.3 Field Quality Control Procedures

A quality control (QC) program will be implemented to ensure that collected data are accurate and precise in order to effectively characterize both the magnitude and variations in ambient conditions at the monitoring stations. Complete documentation of the results of routine operations and QC aspects of the program, including log notes, calibration forms, and certifications, will be maintained on file. Table 4 presents the frequency and type of field QC samples. Key elements of the routine field QC program will include the following:

- Routine visits, approximately every two to three hours during the workday, to each sampling station over the sampling period to check sampler pump flow rates, verify operation and sample conditions, and note ambient conditions that could affect the accuracy or representativeness of the sample
- Collection of field blanks at a frequency of one sample per week while equipment is in operation (Table 4)
- Monthly or weekly flow rate verification using an external National Institute for Standards and Technology-traceable flow meter (Table 5)
- Calibration of the sampling pumps and flow devices
- Routine preventative maintenance of equipment components

Air sampling will be conducted at upwind and downwind locations located on Parcel G. Precision will be measured with every sample batch through laboratory duplicates from submitted samples (Table 4).

Additionally, a collocated real-time dust monitor will be placed at the southwest monitoring site, with the other instruments rotating through on a regular basis. This real-time dust monitor will be available if a replacement instrument is needed, if another location needs to be sampled, or if an instrument malfunctions. The criteria for evaluation of PM<sub>10</sub> precision between real-time instruments will be  $\pm 30$  percent over the sampling day.

#### 3.3.1 Calibration Flow Rates and Maintenance

The flow rate of the air samplers will be calibrated to establish traceability of the field measurement. Calibrations will follow the guidelines specified in 40 CFR, Part 50, Section 9.3 and Section 2.6 of EPA *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods* (1998). Calibration procedures will follow manufacturer's instructions and will utilize manufacture calibration kits as applicable. Table 5 describes field sampler calibration, monitoring and maintenance.

Air-sampling and dust-monitoring equipment will be inspected daily (workdays) before operation and water vapor, insects, or other accumulation that may affect monitoring will be removed. If necessary and at the discretion of the on-site air-monitoring personnel, the air-sampling and dust-monitoring

equipment may need to be shut down and protected during precipitation events. These events are deemed necessary due to safety concerns and equipment integrity issues. To protect against adverse weather, the high-volume air samplers used for collecting PM<sub>10</sub> and TSP are housed in rugged all-weather shelters. Real-time dust monitors (DustTrak™ II or equivalent) are housed in environmental enclosures to protect against the elements. Equipment shutdowns will be noted in the daily production report. Shutdowns of air-sampling stations, real-time dust monitors, or individual units being inoperable will not preclude removal activities at the associated work site. If work proceeds with inoperable air-sampling stations, real-time dust monitors, or individual units the Navy will be notified. If instruments are inoperable for more than a week, the regulatory agencies will be notified. Shutdowns will be kept to a minimum and will be described in air quality monitoring reports.

### 3.3.2 Sample Documentation, Custody and Field Logs

Field logs will be used to properly record information after collecting the samples. Appropriate field data, such as date, time, sample identification, calibration data, sample location, ambient temperature and pressure, and additional information or observations that could influence analyses of the results will be entered on the field logs. Attachment 2 provides field forms.

Samples shipped off site for analysis will be documented on a Chain of Custody Form following the standard procedure described in the Sampling and Analysis Plan Addendum (Appendix B of this Work Plan Addendum). Standard sample ID format will be applied to air quality monitoring samples collected throughout the project duration. The sample IDs are assigned using the following format:

Parcel ID -TEST Date-Station ID

*Examples*

PG-TSP120120-1UPWIND

PG-PM10120120-17DOWNWIND

## 4.0 DATA REVIEW AND REPORTING

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Dust-control activities, wind data, air-monitoring equipment shutdowns, and PDR results will be documented during construction activities and included in the Daily Contractor Production Reports. The APTIM Project Chemist will review off-site laboratory results for compliance with laboratory specified bias, precision, and accuracy requirements. The data review will be in follow the general guidelines in the *National Functional Guidelines for Inorganic Superfund Methods Data Review* (EPA, 2017). The Project Chemist in conjunction with Project Task Lead will compile laboratory and field data for reporting to the Navy Remedial Project Manager. The Navy will report dust-control activities, wind data, and PDR results to EPA and DTSC on a weekly basis. Air-sampling reports will be prepared as analytical results are received from the laboratory and electronically submitted biweekly (depending on the receipt of analytical data) to the Navy. The Navy will provide the reports to EPA and DTSC on a biweekly basis and will post them to the Former Hunters Point Naval Shipyard website as they are available. The air-sampling report will include a brief summary of laboratory data quality, will identify any QC outliers and will include a statement of data usability for project decision. Laboratory reports will be included in the remedial action completion report.

## 5.0 REFERENCES

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Aptim Federal Services LLC, 2019, *Final Accident Prevention Plan, Radiological Investigation, Survey, and Reporting, Parcel G, Former Hunters Point Naval Shipyard, San Francisco, California.*

Aptim Federal Services LLC, 2020, *APTIM Management System.*

Bay Area Air Quality Management District, 2015, *Particulate Matter and Visible Emissions*, 6 301 Ringelman No. Limitation, 6 302 Opacity Limitation, and 6 305 Visible Particles, October.

California Department of Toxic Substances Control, 2019, *Human and Ecological Risk Office (HERO) Memorandum, Draft Dust Action Levels for Parcel G, Hunters Point Naval Shipyard, San Francisco, California*, March 5.

CH2M Hill, Inc., 2019, *Final Parcel G Removal Site Evaluation Work Plan, Former Hunters Point Naval Shipyard, San Francisco, California*, June.

National Institute for Occupational Safety and Health, 1994, *NIOSH Manual of Analytical Methods, Method 7400*, August.

Tetra Tech EC, 2010, *Final Basewide Dust Control Plan, Revision 1, Hunters Point Shipyard, San Francisco, California*, November 29.

U.S. Environmental Protection Agency, 1998, *Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods.*

U.S. Environmental Protection Agency, 1999, *Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air.*

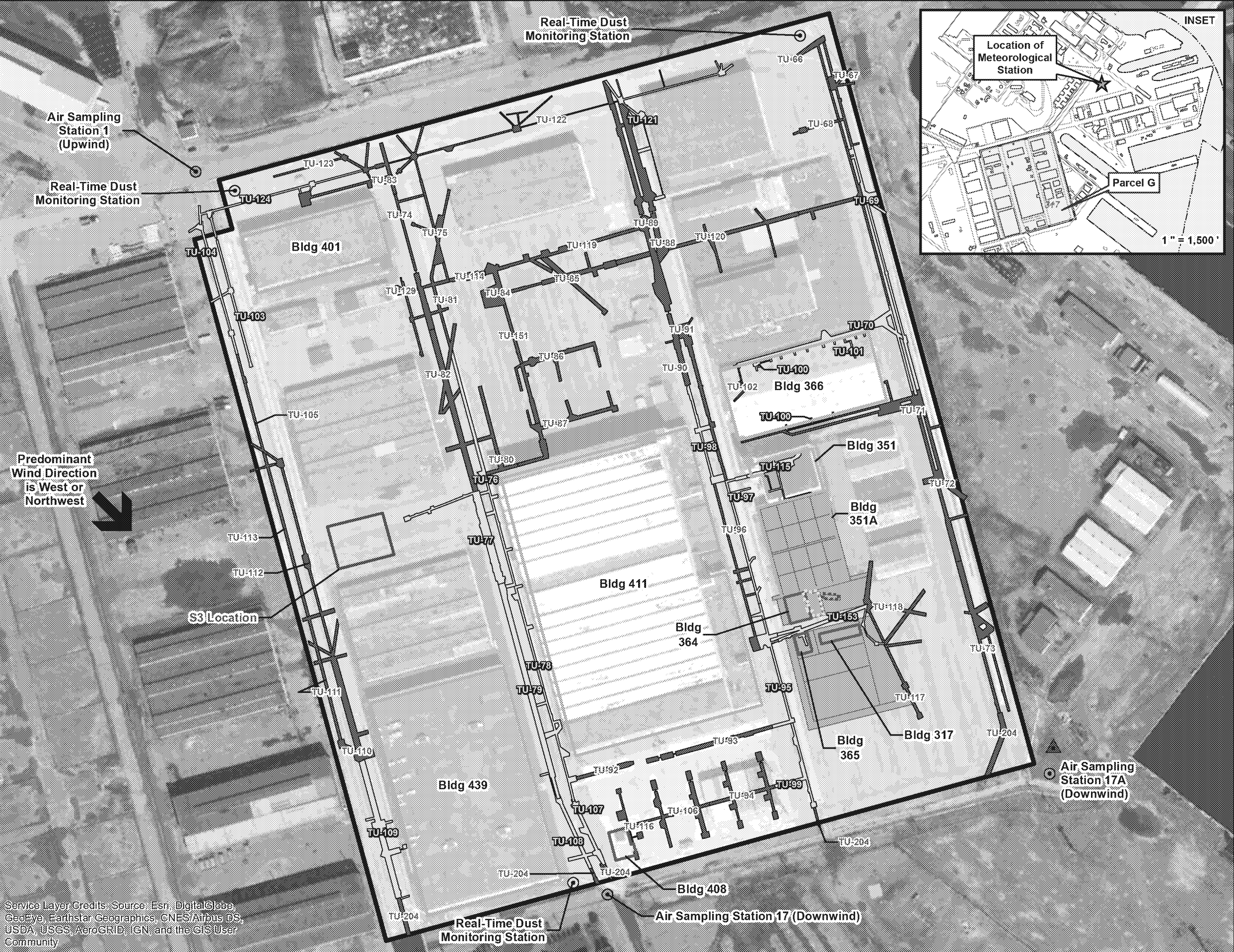
U.S. Environmental Protection Agency, 2017, *National Functional Guidelines for Inorganic Superfund Methods Data Review*, January.

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## Figures

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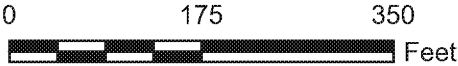
- Air Sampling Station (Worker Action Levels for COCs [Table 1] and Public Action Levels for ROCs [Table 2])
- Real-Time Dust Monitoring Station (Public Action Level for Dust [PM10] [Table 1])
- Windsock
- Parcel G Boundary
- Impacted Building
- Impacted Demolished Building
- Phase 1 Trench Unit
- Phase 2 Trench Unit
- Survey Unit
- Liquid Waste Transfer System Excavation Limits (to 10 ft bgs)
- Peanut Spill Excavation Limits (to 2 ft bgs)
- S3 Location

Notes:

- 1) Air sampling will be performed at two stations, one upwind (Station 1) and one downwind (Station 17 or 17A). Air sampling station locations represent potential locations and may be modified to accomodate changing site conditions and/or wind direction.
- 2) Additional dust monitoring locations may be established based on field activities.
- 3) COC - Chemical of Concern.
- 4) ROC - Radionuclide of Concern.
- 5) Location of S3 may change based on actual site conditions and professional judgment.



1 inch = 175 feet



U.S. Department of the Navy  
BRAC PMO West  
San Diego, California

FIGURE 1  
PARCEL G  
AIR SAMPLING AND DUST  
MONITORING LOCATIONS

INVESTIGATION, SURVEY, AND REPORTING AT PARCEL G  
HUNTERS POINT NAVAL SHIPYARD, SAN FRANCISCO, CA

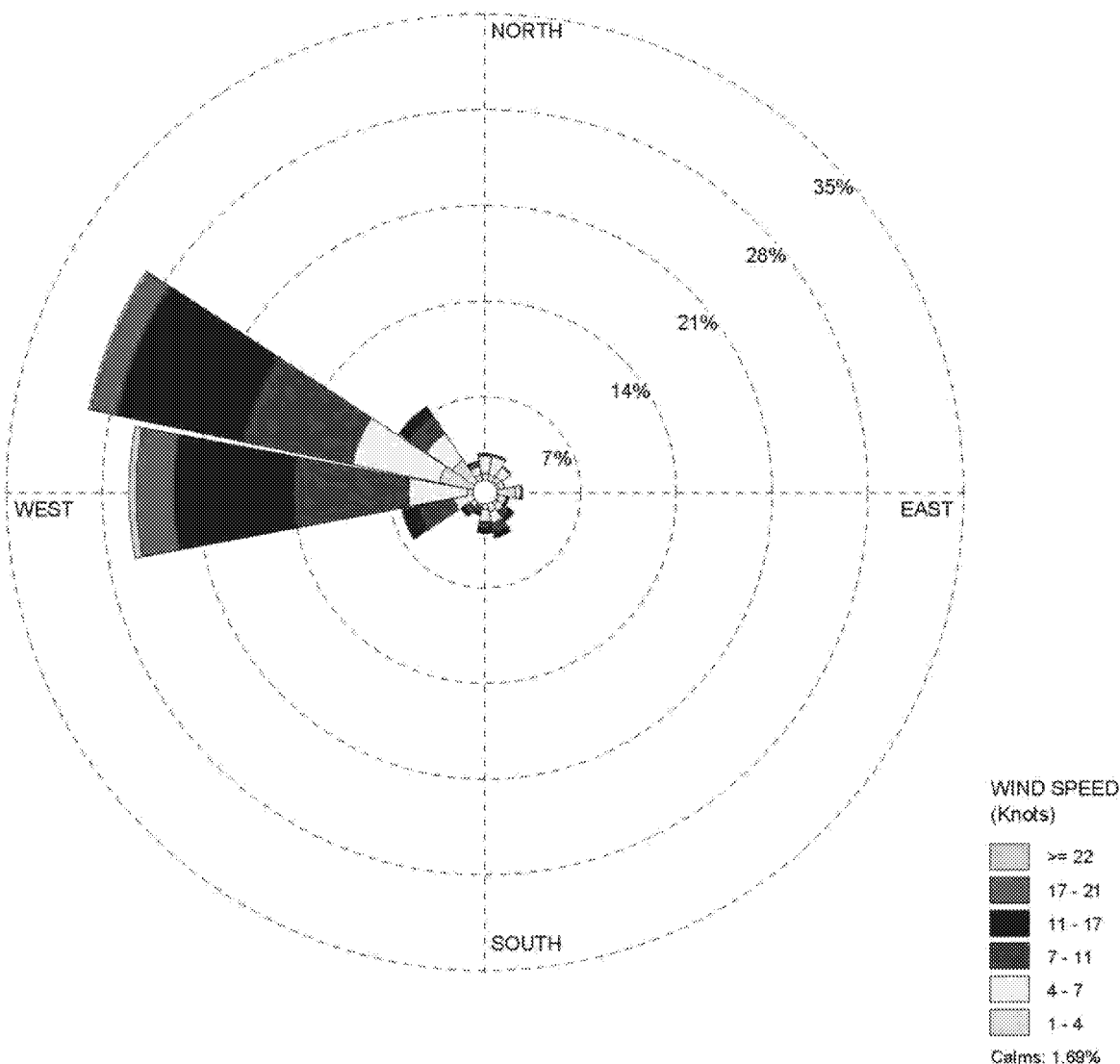
Service Layer Credits: Source: Esri, DigitalGlobe, GeoEye, Earthstar Geographics, CNES/Airbus DS, USDA, USGS, AeroGRID, IGN, and the GIS User Community

WIND ROSE PLOT:

**Parcel E Met Station**  
**August 1, 2004 - May 31, 2010**

DISPLAY:

**Wind Speed**  
**Direction (blowing from)**



COMMENTS:

**Hunters Point Shipyard,  
San Francisco, CA**

DATA PERIOD:

**2004-2010**  
**Jan 1 - Dec 31**  
**00:00 - 23:00**

COMPANY NAME:

**Innovative Technical Solutions, Inc.**

MODELER:

**Brett Womack**

CALM WINDS:

**1.69%**

TOTAL COUNT:

**6325 hrs.**

AVG. WIND SPEED:

**8.14 Knots**

DATE:

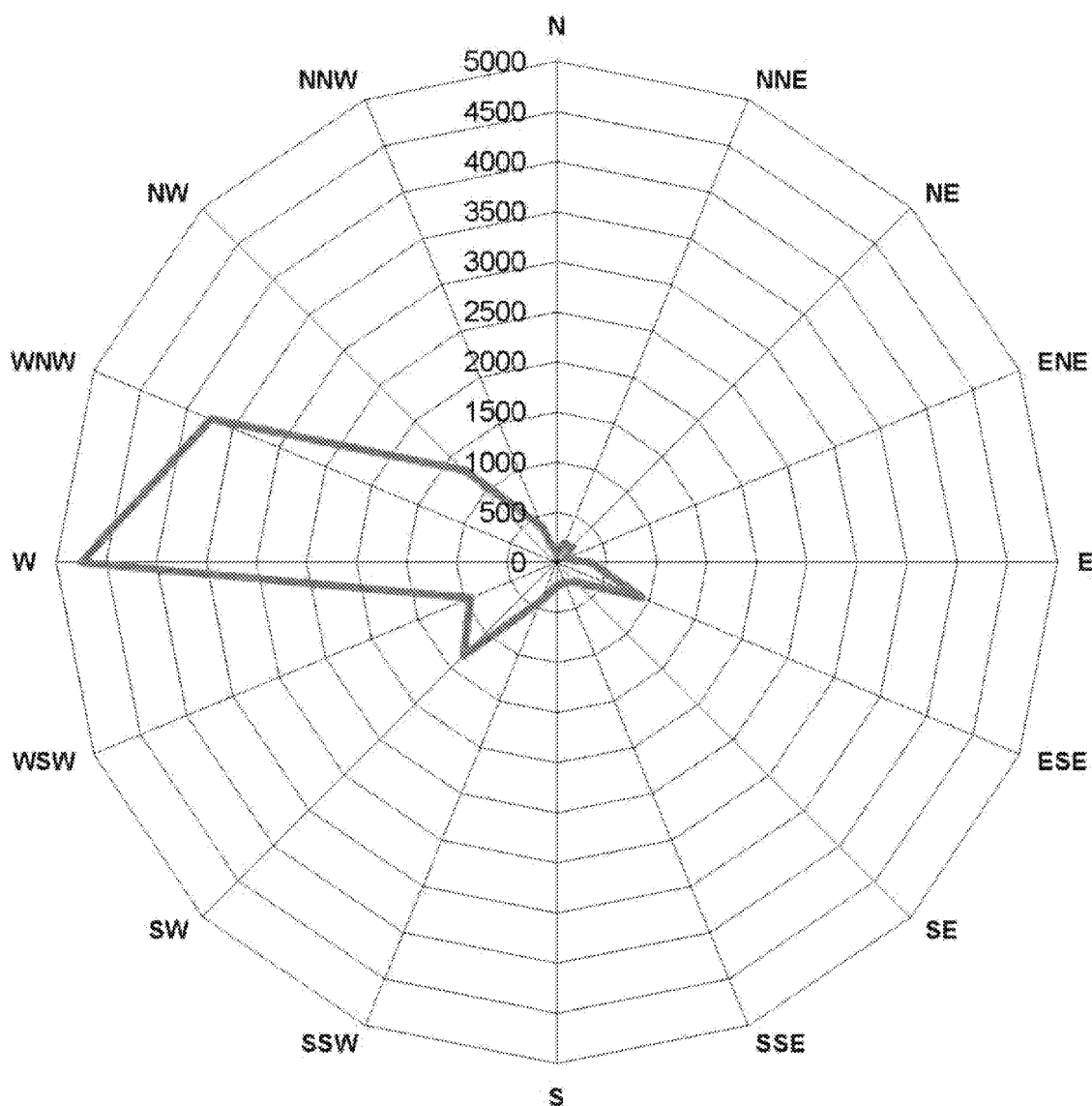
**6/1/2010**

**U.S. Department of the Navy**  
**BRAC PMO West**  
**San Diego, California**

**FIGURE 2**  
**WIND ROSE**  
**PARCEL E**

INVESTIGATION, SURVEY, AND REPORTING AT PARCEL G  
HUNTERS POINT NAVAL SHIPYARD, SAN FRANCISCO, CA

WRPLOT View - Lakes Environmental Software



— Number of events

**Notes:**

1. The wind rose represents wind direction data recorded at an onsite meteorological station for 2015. Data have been collected continuously since construction work began at Hunters Point; 15,000 data points were collected in 2015.
2. The predominant wind direction is from the West or West-Northwest.

**Reference:**

Geosyntec Consultants, 2016, Hunters Point Artists Parcel Project, Navy Parcels B-1, C, and UC-2, Hunters Point Shipyard, San Francisco, California.

U.S. Department of the Navy  
BRAC PMO West  
San Diego, California

**FIGURE 3**  
**WIND ROSE**  
**PARCELS B-1, C, AND UC-2**

INVESTIGATION, SURVEY, AND REPORTING AT PARCEL G  
HUNTERS POINT NAVAL SHIPYARD, SAN FRANCISCO, CA

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## Tables

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**Table 1**  
**Dust-Monitoring and Air-Sampling Threshold Criteria**

Contaminant of Concern	Action Level	Sample Time Interval	Basis	Reporting Limit <sup>1</sup>	Response
Real-time Dust Monitoring					
Real-time dust monitoring (PM10)	50 µg/m <sup>3</sup>	24 hours	DTSC HERO developed action level <sup>2</sup> (residents and public receptors)	1.0 µg/m <sup>3</sup>	Dust control measures will be implemented if real-time dust monitoring data has sustained exceedances of 50 µg/m <sup>3</sup> over a 15-minute interval.
Filter-Based Air Sampling					
TSP	500 µg/m <sup>3</sup>	24 ± 1 hours <sup>3</sup>	Basewide HPNS level chosen to minimize overall permissible dust release from sites	0.0003 µg/m <sup>3</sup>	<p>Compare data to real-time dust monitoring, wind speed data, and Daily Construction Production Reports to determine cause of exceedances.</p> <p>If exceedances are project-related, additional dust control measures will be implemented.</p> <p>If exceedances are not project-related, Navy will evaluate appropriateness of monitoring locations, sampling methods, and sampling frequency.</p> <p>Navy will notify regulatory agencies.</p>
Pb	50 µg/m <sup>3</sup>	24 ± 1 hours <sup>3</sup>	Cal/OSHA PEL (on-site workers)	0.020 µg/m <sup>3</sup>	
Mn	200 µg/m <sup>3</sup>	24 ± 1 hours <sup>3</sup>	Cal/OSHA PEL (on-site workers)	0.020 µg/m <sup>3</sup>	
PM10	5,000 µg/m <sup>3</sup>	24 ± 1 hours <sup>3</sup>	Cal/OSHA PEL <sup>4</sup> (on-site workers)	0.0003 µg/m <sup>3</sup>	
PM10	50 µg/m <sup>3</sup>	24 ± 1 hours <sup>3</sup>	DTSC HERO developed action level <sup>2</sup> (residents and public receptors)	0.0003 µg/m <sup>3</sup>	
Asbestos	0.1 fiber per cubic centimeter	24 ± 1 hours <sup>3</sup>	Cal/OSHA PEL (on-site workers)	0.005 fiber per cubic centimeter	

**Table 1 (continued)**  
**Dust-Monitoring and Air-Sampling Threshold Criteria**

*Notes:*

<sup>1</sup> Reporting limit is based on volume of air collected and will vary, reporting limit shown is typical for normal operation day.

<sup>2</sup> Action level for dust (PM<sub>10</sub>) was developed by DTSC HERO, is based on a 24-hour average concentration, and is protective for all Parcel G non-radiological contaminants of concern (DTSC, 2019).

<sup>3</sup> If there have been no sustained exceedances of 50 µg/m<sup>3</sup> over a 15-minute interval from the real-time dust monitors on a Friday (or day preceding a holiday), samples may be collected at the end of that workday.

<sup>4</sup> Cal/OSHA PEL for particulates not otherwise regulated (respiratory) used for PM<sub>10</sub>.

µg/m <sup>3</sup>	microgram per cubic meter
Cal/OSHA	California Occupational Safety and Health Administration
DTSC	California Department of Toxic Substances Control
EPA	U.S. Environmental Protection Agency
HERO	Human and Ecological Risk Office
HPNS	Former Hunters Point Naval Shipyard
mg/m <sup>3</sup>	milligram per cubic meter
Mn	manganese
Navy	U.S. Department of the Navy
Pb	lead
PEL	permissible exposure limit
PM <sub>10</sub>	particulate matter smaller than 10 microns in diameter
TSP	total suspended particulates

**Table 2**  
**Radionuclides of Concern Airborne Concentration Action Levels**

ROC	ROC Class	Radiations	Worker <sup>1</sup>	Off-site Receptor <sup>2</sup>	Laboratory or Field Reporting Limit <sup>3</sup> (μCi/mL)
			10% DAC (μCi/mL)	Effluent Concentration (μCi/mL)	
Laboratory Measurements					
Cesium-137	D, all compounds <sup>4</sup>	Gamma, beta	6.0E-9	4.00E-11	2.04E-16
Plutonium-239 <sup>5</sup>	Y, PuO <sub>2</sub> <sup>5</sup>	Alpha	7.0E-13	4.00E-15	1.02E-15
Radium-226	W, all compounds <sup>4</sup>	Gamma, alpha	3.0E-11	1.80E-13	1.14E-14
Strontium-90	Y, all insoluble compounds and SrTiO <sub>3</sub> <sup>4</sup>	Beta	2.0E-10	1.20E-12	8.17E-16
Thorium-232 <sup>6</sup>	Y, oxides and hydroxides <sup>6</sup>	Gamma, alpha	1E-13	1.20E-15	8.17E-16
Uranium-235 <sup>7</sup>	D, UF <sub>6</sub> , UO <sub>2</sub> F <sub>2</sub> , UO <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> <sup>7</sup>	Alpha	6E-11	6.00E-13	8.17E-16
Field Measurements					
Gross alpha/beta on-site measurements with Ludlum Model 3030 (or equivalent)	N/A	Alpha Beta	1.0E-13 <sup>8</sup> 2.0E-10 <sup>8</sup>	N/A	4.8E-14 4.6E-13

*Notes:*

<sup>1</sup> The guideline values were determined using 10 percent of the DAC provided in the 10 CFR, Part 20, Appendix B, and specified in the Final Basewide Dust Control Plan, Revision 1, Hunters Point Shipyard, San Francisco, California (Tetra Tech EC, Inc., 2010) and the Final Parcel G Removal Site Evaluation Work Plan, Former Hunters Point Naval Shipyard, San Francisco, California (CH2M Hill, Inc., 2019), as noted.

<sup>2</sup> The effluent concentration values were determined using the 10 CFR, Part 20, Appendix B, Table 2, Column 1 adjusted from 50 mrem per year to a maximum annual exposure of 10 mrem per year at the receptor. Results may be evaluated using 40 CFR Appendix E to Part 61 to demonstrate compliance with the National Emission Standards for Hazardous Air Pollutants (40 CFR Part 61).

<sup>3</sup> Reporting limit is based on volume of air collected and will vary, reporting limit shown is typical for normal operation day.

<sup>4</sup> The ROC class was determined in the Final Basewide Dust Control Plan, Revision 1, Hunters Point Shipyard, San Francisco, California (Tetra Tech EC, Inc., 2010).

**Table 2 (continued)**  
**Radionuclides of Concern Airborne Concentration Action Levels**

*Notes (continued):*

<sup>5</sup> Plutonium-239 is a ROC for the Former Buildings 317/364/365 Site, Building 351A crawl space, and associated trench units 95, 97, 115, 117, 118, and 153 only. Class Y plutonium oxide is the appropriate radionuclide class as plutonium at HPNS has oxidized due to environmental weathering for many years.

<sup>6</sup> Thorium-232 is a ROC for the Building 351A crawl space and associated trench units 97, 107, and 115 only. Class Y Thorium Oxide is the appropriate radionuclide class as thorium at HPNS has oxidized due to environmental weathering for many years.

<sup>7</sup> Uranium-235 is a ROC for the Former Buildings 317/364/365 Site and associated trench units 95, 117, 118, and 153 only. The DAC for uranium-235 was provided in the Final Parcel G Removal Site Evaluation Work Plan, Former Hunters Point Naval Shipyard, San Francisco, California (CH2M Hill, Inc., 2019). Ten percent of the DAC is used to be conservative.

<sup>8</sup> The most conservative concentration will be used for alpha and beta on-site measurements. The Ludlum Model 3030 (or equivalent) counts filters in units of counts per minute. Using the alpha/beta air-monitoring field form (Attachment 2), the counting results are converted to units of  $\mu\text{Ci/mL}$ . The conversion calculation is based on sample run time, flow rate, background, efficiency, and counts per minute.

$\mu\text{Ci/mL}$	microcuries per milliliter (activity)
%	percent
CFR	Code of Federal Regulations
D	daily
DAC	derived airborne concentration
HPNS	Former Hunters Point Naval Shipyard
mrem	millirem
N/A	not applicable
$\text{PuO}_2$	plutonium oxide
ROC	radionuclide of concern
W	weekly
Y	yearly



**Table 3**  
**Air-Sampling and Dust-Monitoring Frequency and Sample Collection Methods**

Sampling Scenario	Type of Analysis	Sampling Method <sup>1</sup>	Frequency
Excavation and soil handling (upwind and downwind)	TSP	40 CFR, Part 50, Appendix B	One sample per 24 hours <sup>3</sup>
	Metals (Mn, Pb)	40 CFR, Part 50, Appendix B	One sample per 24 hours <sup>3</sup>
	PM10	40 CFR, Part 50, Appendix J	One sample per 24 hours <sup>3</sup>
	Asbestos	NIOSH Method 7400	One sample per 24 hours <sup>3</sup>
	ROCs	D2006-4550-011 <sup>2</sup>	One sample per work week <sup>4</sup>
	Real-time dust monitoring	DustTrak™ II or equivalent	Continuous daily during work <sup>5</sup>
Placement of backfill and site restoration (upwind and downwind)	TSP	40 CFR, Part 50, Appendix B	One sample per 24 hours <sup>3</sup>
	Metals (Mn, Pb)	40 CFR, Part 50, Appendix B	One sample per 24 hours <sup>3</sup>
	PM10	40 CFR, Part 50, Appendix J	One sample per 24 hours <sup>3</sup>
	Asbestos	NIOSH Method 7400	One sample per 24 hours <sup>3</sup>
	ROCs	D2006-4550-011 <sup>2</sup>	One sample per work week <sup>4</sup>
	Real-time dust monitoring	DustTrak™ II or equivalent	Continuous daily during work <sup>5</sup>

**Notes:**

<sup>1</sup> Sampling method to be employed for emissions analysis or industry-recognized equivalent. Laboratory standard operating procedures for analysis are included in Table 4 and provided in Attachment 3.

<sup>2</sup> D2006-4550-011, "Operation and Use of Portable Instruments at Hunters Point Naval Shipyard" (APTIM, 2020), is a work instruction used for radiological air-sampling activities by APTIM and is provided in Attachment 3.

<sup>3</sup> If there have been no sustained exceedances of 50 µg/m<sup>3</sup> over a 15-minute interval from the real-time dust monitors on a Friday (or day preceding a holiday), samples may be collected at the end of that workday.

<sup>4</sup> A work week will span approximately 104 hours.

<sup>5</sup> The length of a workday may range from a minimum of four hours up to 14 hours.

APTIM	Aptim Federal Services, LLC
CFR	Code of Federal Regulations
Mn	manganese
NIOSH	National Institute for Occupational Safety and Health
Pb	lead
PM10	particulate matter smaller than 10 microns in diameter
ROC	radionuclide of concern
TSP	total suspended particulates

**Table 4**  
**Field and Laboratory Quality Control Samples**

Parameter	Blank (Bias Contamination)	Laboratory/Field Control Sample (Accuracy)	Field/Laboratory Duplicate (Precision)	SOP Reference	Laboratory/ Organization
Asbestos PCM 7400/NIOSH 7400	Field filter blanks submitted with each sample group  Results < reporting limit; see SOP if fibers detected in blank	Blind Daily Reference Slide per analyst; control limits vary based on slide submitted; Analyst must fall within limits	Laboratory precision is calculated per analyst based on last 20 reference slides. Standard deviation $\leq 0.45$	EMSL SOP -ASB-SOP-300, "Asbestos and Other Fibers by PCM 7400," NIOSH 7400 Issue 3	LA Testing 5431 Industrial Drive Huntington Beach, CA  Project Manager: Terry Chang 714 895 5494
Dust (measured as PM10)	Internal Self Zero twice/day	Weekly flow rate check with external NIST traceable flow calibrator; 3 L/min tolerance $\pm 5\%$	Field precision is calculated using one (1) collocated dust monitor  RPD $\leq 30\%$ for detections above 15 $\mu\text{g}/\text{m}^3$	Manufacturers operation manual and Section 3.2.4.2	EquipCo Rentals PO Box 5606 Concord, CA Representative: Jason Miller 888.234.5678  APTIM Equipment Services 16406 E US Route 224, Bay 5 Findlay, OH Representative: Michael Hindall 419.425.6054
PM10	Weekly Field Filter Blank—Difference in initial and final concentration $\pm 5 \mu\text{g}/\text{m}^3$  Laboratory Filter Blank- Daily per analytical batch Results < reporting limit	Analytical balance accuracy check per SOP	Laboratory Duplicate  Daily per analytical Batch  RPD $\leq 10\%$	Eurofins-Calscience SOP-M791, "40 CFR, Part 50 Appendix J, PM-10 Gravimetric Measurements"  and  "Total Suspended Particulates in Air"	Eurofins Calscience 7440 Lincoln Way Garden Grove, CA  Project Manager: Terry Chang 714 895 5494

**Table 4 (continued)**  
**Field and Laboratory Quality Control Samples**

Parameter	Blank (Bias Contamination)	Laboratory/Field Control Sample (Accuracy)	Field/Laboratory Duplicate (Precision)	SOP Reference	Laboratory/ Organization
TSP	Weekly Field Filter Blank—Difference in initial and final concentration $\pm 5 \mu\text{g}/\text{m}^3$  Laboratory Filter Blank- Daily per analytical batch Results < reporting limit	Analytical balance accuracy check per SOP	Laboratory Duplicate  Daily per analytical Batch  RPD $\leq 25\%$		Eurofins Calscience 7440 Lincoln Way Garden Grove, CA  Project Manager: Terry Chang 714 895 5494
Metals (Pb/Mn) on TSP filter EPA 6010	Laboratory Filter Blank  Daily per analytical batch  Results < reporting limit	Daily per analytical batch  Pb % Recovery: 80%–120%  Mn % Recovery: 80%–120%	Laboratory Duplicate  Daily per analytical batch  RPD $\leq 20\%$	Eurofins-Calscience  SOP-M623, “40 CFR, Part 50, Appendix G, Determination of Lead in Suspended Particulate Matter Collected from Ambient Air”  and  SOP-M601, “EPA Method 6010B, ICP, AES”	Eurofins Calscience 7440 Lincoln Way Garden Grove, CA  Project Manager: Terry Chang 714 895 5494
Gross alpha/beta on-site measurements with Ludlum Model 3030 (or equivalent)	Field filter cartridge blank at the start and end of sample counting  Results $\leq$ Background	Daily response source check  Alpha Check Source: $^{230}\text{Th}$  Beta Check Source: $^{99}\text{Tc}$  +/- 20% of known	Field Replicate Reading  One per 10 field samples  RPD $\leq 30\%$  (for results > Background)	APTIM Work Instruction D2006-4550-011, “Operation and Use of Portable Instruments at Hunters Point Naval Shipyard”	APTIM Equipment Services 16406 E US Route 224, Bay 5 Findlay, OH Representative: Michael Hindall 419 425 6054

**Table 4 (continued)**  
**Field and Laboratory Quality Control Samples**

Parameter	Blank (Bias Contamination)	Laboratory/Field Control Sample (Accuracy)	Field/Laboratory Duplicate (Precision)	SOP Reference	Laboratory/ Organization
Off-Site Gamma Spec Laboratory Analysis: $^{137}\text{Cs}$	Field Filter Blank  Daily per analytical batch  No analytes detected greater than MDC	One per analytical batch  Recovery Limits: $^{137}\text{Cs}$ : 75–125% $^{60}\text{Co}$ : 75–125% $^{241}\text{Am}$ : 75–125%	One per analytical batch  RPD $\leq 25\%$	Eurofins-TestAmerica SOP ST-RC-0004, “Filter Prep”  and SOP ST-RD-0102, “Gammavision® Analysis,” 3/22/19, Revision 18	TestAmerica St. Louis 13715 Rider Trail N, Earth City, MO  Project Manager: Rhonda Ridenhower 314 298 8566
Off-Site Laboratory Analysis: $^{90}\text{Sr}$	Field Filter Blank  Daily per analytical batch  No analytes detected greater than MDC	One per analytical batch  Recovery Limits: 75–125%	One per analytical batch  RPD $\leq 25\%$	Eurofins-TestAmerica SOP ST-RC-0004, “Filter Prep”  and SOP ST-RD-0403, “Low Background Gas Flow Proportional Counting System Analysis”	TestAmerica St. Louis 13715 Rider Trail N, Earth City, MO  Project Manager: Rhonda Ridenhower 314 298 8566
Off-Site Alpha Spec Laboratory Analysis: $^{239}\text{Pu}$ , $^{226}\text{Ra}$ , $^{232}\text{Th}$ , $^{235}\text{U}$	Field Filter Blank  Daily per analytical batch  No analytes detected greater than MDC	One per analytical batch  Recovery Limits: $^{241}\text{Am}$ : 75–125% $^{238}\text{Pu}$ : 80–127% $^{239/240}\text{Pu}$ : 75–125% $^{238}\text{U}$ : 75–125% $^{226}\text{Ra}$ : 75–125% $^{232}\text{Th}$ : 75–125%	One per analytical batch  RPD $\leq 25\%$	Eurofins-TestAmerica SOP ST-RC-0004, “Filter Prep”  and SOP ST-RD-0210, “Alpha Spectroscopy Analysis”	TestAmerica St. Louis 13715 Rider Trail N, Earth City, MO  Project Manager: Rhonda Ridenhower 314 298 8566

**Table 4 (continued)**  
**Field and Laboratory Quality Control Samples**

*Notes:*

*Attachment 3 provides Eurofins-Calscience SOPs, Eurofins-TestAmerica ST-RC-004, and APTIM Work Instruction D2006-4550-011, "Operation and Use of Portable Instruments at Hunters Point Naval Shipyard." Other SOPs for Eurofins-TestAmerica for off-site analysis (if needed) are provided in Attachment 2 to the Sampling and Analysis Plan Addendum (Appendix B of this Work Plan Addendum).*

%	percent
≤	less than or equal to
<sup>241</sup> Am	americium-241
<sup>60</sup> Co	cobalt-60
<sup>137</sup> Cs	cesium-137
<sup>238</sup> Pu	plutonium-238
<sup>239</sup> Pu	plutonium-239
<sup>226</sup> Ra	radium-226
<sup>90</sup> Sr	strontium-90
<sup>232</sup> Th	thorium-232
<sup>235</sup> U	uranium-235
CFR	Code of Federal Regulations
EMSL	EMSL Analytical, Inc. Management
EPA	U.S. Environmental Protection Agency
L/min	liters per minute
MDC	minimum detected concentration
Mn	manganese
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute for Standards and Technology
Pb	lead
PCM	phased contrast microscopy
PM10	particulate matter smaller than 10 microns in diameter
RPD	relative percent difference
SOP	standard operating procedure
TSP	total suspended particulate

**Table 5**  
**Air-Sampling Unit Flow Checks and Controls**

Units/Flow Rate	Flow Rate	Drift	Unit taken out of service
High-volume Air Sampler (TSP and PM <sub>10</sub> ) (39 to 60 cubic feet per minute)	Set/check integrated flow meter at start of day and end of day  Verified flow monthly, or anytime the unit is moved, using external NIST-traceable flow controller  Flow rates recorded on sample collection log	Verified quarterly or any time the unit is moved, or for each unit using manufacture calibration kits  Flow rate verified with external NIST-traceable flow controller monthly (or more frequently if deemed necessary)  Calibration recorded in the Calibration Logbook	When drift cannot be corrected, or calibration fails or expires  PM <sub>10</sub> sampler taken out of service quarterly for cleaning the PM <sub>10</sub> size selective sampling device
Asbestos Low-volume Air Sampler 25-millimeter cassette: 2 to 4 liters per minute (400-liter minimum) 37-millimeter cassette: 2 to 10 liters per minute (3,000-liter minimum)	Set/checked at start and end of each day  Verified monthly, or anytime the unit is moved, using NIST traceable flow controller  Flow rates recorded on sample collection log	Verified quarterly or any time the unit memory is full, using manufacture calibration kits  Calibration recorded in the Calibration Logbook	When it fails calibration
ROCs Low-volume Samplers (60 liters per minute)	Set/checked at start of day with NIST traceable calibrated rotameter  Flow rates recorded in the ROC Air Sample Logbook and on the sample collection envelope	Verified daily upon setup (with rotameter)  Calibration performed yearly, or after repair; recorded in the ROC Air Sample Logbook	When unable to achieve 60 liters per minute, requires repair, or manufacture calibration is due (yearly)
DustTrak™ II PM <sub>10</sub> Real-time Dust Monitor	Daily flow rates are internally continuously monitored	Verified weekly $\pm$ 5 percent setpoint/ DustTrak™ II Operation and Service Manual  Recalibrate as needed	Check power supply  When zero control cannot be maintained or per manufacturers guidelines

*Notes:*

NIST                      *National Institute for Standards and Technology*  
PM<sub>10</sub>                      *particulate matter smaller than 10 microns in diameter*  
ROC                      *radionuclide of concern*  
TSP                      *total suspended particulate*

## Attachment 1 Crosswalk

UFP SAP Worksheet #	Information	Crosswalk to Related Information in Work Plan Addendum
1, 2	Title and Approval Page	DMP Title and Approval Page
3, 5	Distribution List/Project Organizational Chart	SAP WSs 3 and 5, DMP Table 4
4, 7	Personnel Qualifications/Responsibilities and Sign-off Sheet	SAP WSs 4 and 7
6	Communication Pathways	SAP WS 6
7	Personnel Responsibilities Table	SAP WS 7, the Project Chemist and the PRSO/Radiological Supervisor are responsible for air monitoring
8	Special Personnel Training Requirements Table	SAP WS 8 and DMP Section 3.0
9	Project Planning Session Summary Sheet	Meeting Summary and Next Steps: March 31, 2020, Discussion on EPA General Comments #30 and #34 Work Plan Addendum
10	Conceptual Site Model	SAP WS 10
11	Project/Data Quality Objectives	<ul style="list-style-type: none"> <li>• DQO Step 1—Problem Statement: DMP Section 1.0, Paragraph 1, provides the problem statement</li> <li>• DQO Step 2—Objective: DMP Section 1.0, Paragraph 3, states air monitoring is performed to ensure worker safety and provide reasonable assurance of the protection of the surrounding residents</li> <li>• DQO Step 3—Inputs to Objective: DMP Section 1.0, Paragraph 3, identifies data inputs</li> <li>• DQO Step 4—Study Boundaries: DMP Figure 1 shows the study boundaries</li> <li>• DQO Step 5—Decision Rules: DMP Section 3.0, Paragraph 3, DMP Section 3.2.4.1, Paragraph 2, DMP Section 3.2.4.2, Paragraph 1, DMP Section 3.2.5, Paragraph 1, and DMP Table 1 identify the decision rules</li> <li>• DQO Step 6—Performance Criteria: Sample data will be compared to DMP Tables 1, 2, and 4</li> <li>• DQO Step 7—Plan for Obtaining Data: DMP Sections 2.0 and 3.2 provide the plan for obtaining air monitoring data</li> </ul>
12	Field Quality Control Samples (per NAVFAC TIER 1 SAP)	DMP Table 4
13	Secondary Data Criteria and Limitations Table	DMP Sections 2.2.10 and 3.1
14	Summary of Project Tasks	DMP Sections 2.0, 3.0, and 4.0
15	Reference Limits and Evaluation Tables (per NAVFAC TIER 1 SAP)	DMP Tables 1 and 2



UFP SAP Worksheet #	Information	Crosswalk to Related Information in Work Plan Addendum
16	Project Schedule/Timeline Table	SAP WS 16
17	Sampling Design and Rationale	DMP Section 2.2.10, and 3.0 through 3.2.5
18	Location-Specific Sampling Methods/SOP Requirements Table	DMP Figure 1 and Tables 3 and 4
19	Field Sampling Requirements Table	DMP Section 3.2 (including 3.2.1 through 3.2.5)
20	Field QC Sample Summary Table	DMP Table 4
21	Project Sampling SOP References Table	DMP Tables 3, 4, and Attachment 3 SAP Attachment 2
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	DMP Table 5
23	Analytical SOP References Table	DMP Table 4 and Attachment 3 SAP Attachment 2
24	Analytical Instrument Calibration Table	DMP Attachment 3 SAP WS 24a
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	SAP WS 25
26, 27	Sample Handling System/Sample Custody Requirements	DMP Section 3.3.2 SAP WSs 26 and 27
28	Laboratory QC Samples Table	DMP Table 4 SAP WSs 28a, 28b, and 28c
29	Project Documents and Records Table	SAP WS 29
30	Analytical Services Table	DMP Table 4
31, 32, 33	Assessments and Corrective Action	SAP WSs 32 and 33
34, 35, 36	Data Verification and Validation (Steps I and IIa/IIb) Process Table	DMP Section 4.0
37	Usability Assessment	DMP Section 4.0

*Notes:*

DMP	<i>Dust Management and Air Monitoring Plan</i>
DQO	<i>data quality objective</i>
EPA	<i>U.S. Environmental Protection Agency</i>
NAVFAC	<i>Naval Facilities Engineering Command</i>
PRSO	<i>Project Radiation Safety Officer</i>
QC	<i>quality control</i>
SAP	<i>Sampling and Analysis Plan Addendum (Appendix B of this Work Plan Addendum)</i>
SOP	<i>standard operating procedure</i>
WS	<i>worksheet</i>

## Attachment 2 Field Forms



DATE			20
PAGE:	OF		

### AIR MONITORING SAMPLE COLLECTION LOG

PROJECT NAME **CTO 5065 - Parcel G Rework**

PROJECT No. **501197**

SAMPLE TYPE **Air (Grab)**

ANALYSES	CONTAINER	ANALYSES	CONTAINER
<input checked="" type="checkbox"/> TSP	<input checked="" type="checkbox"/> 8x10 Whatman QM-A Quartz Filter	<input type="checkbox"/> PCBs (EPA method TO-04)	<input type="checkbox"/> Polyurethane foam (PUF) trap
<input checked="" type="checkbox"/> PM-10	<input checked="" type="checkbox"/> 8x10 Whatman QM-A Quartz Filter	<input type="checkbox"/> PAHs (EPA 8270-SIM TO-13)	<input type="checkbox"/> Polyurethane foam (PUF) trap
<input checked="" type="checkbox"/> Asbestos (NIOSH 7400)	<input checked="" type="checkbox"/> 0.8µ PCM filter	<input type="checkbox"/> Dioxin (EPA method TO-9A)	<input type="checkbox"/> Polyurethane foam (PUF) trap

**Field Conditions:**

☐ Sunny
 ☐ Overcast
 ☐ Rain
 ☒ Wind
 ☒ Temperature

Precipitation: \_\_\_\_\_ in.
 Wind Gust: \_\_\_\_\_ mph
 Average Temp: \_\_\_\_\_ °F
 Pressure: \_\_\_\_\_ in.

Runtime				Analysis	Station Code		Sample ID	Comments:
Start		Finish			1			
Date	Time	Date	Time					
				TSP			PG-TSP_____ -STA1UPWIND	
				PM-10			PG-PM10_____ -STA1UPWIND	
				Asbestos			PG-ASBESTOS_____ -STA1UPWIND	

Runtime				Analysis	Station Code		Sample ID	Comments:
Start		Finish			17			
Date	Time	Date	Time					
				TSP			PG-TSP_____ -STA17DOWNWIND	
				PM-10			PG-PM10_____ -STA17DOWNWIND	
				Asbestos			PG-ASBESTOS_____ -STA17DOWNWIND	

The monitoring stations will be supplemented with real-time PM10 air monitoring.

PREPARED BY: \_\_\_\_\_



# Hunters Point Naval Shipyard

## APTIM Air Sample Field Log Book

Air Sample Survey #:			
Air Sample Location:			
Start	Date	Time:	Flow:
Stop	Date	Time:	Flow:
Low Vol ID#:		Cal. Due Date:	
Notes:			
RCT (print/sign):		Date:	

Air Sample Survey #:			
Air Sample Location:			
Start	Date	Time:	Flow:
Stop	Date	Time:	Flow:
Low Vol ID#:		Cal. Due Date:	
Notes:			
RCT (print/sign):		Date:	

Air Sample Survey #:			
Air Sample Location:			
Start	Date	Time:	Flow:
Stop	Date	Time:	Flow:
Low Vol ID#:		Cal. Due Date:	
Notes:			
RCT (print/sign):		Date:	

Air Sample Survey #:			
Air Sample Location:			
Start	Date	Time:	Flow:
Stop	Date	Time:	Flow:
Low Vol ID#:		Cal. Due Date:	
Notes:			
RCT (print/sign):		Date:	

## A2-3

## ALPHA/BETA AIR MONITORING DATA

[illegible]

<b>ROC</b>	<b>Worker</b>	<b>Offsite Receptor</b>
<b>Cs-137</b>	<b>6.00E-09</b>	<b>4.00E-11</b>
<b>Pu-239</b>	<b>7.00E-13</b>	<b>4.00E-15</b>
<b>Ra-226</b>	<b>3.00E-11</b>	<b>1.80E-13</b>
<b>Sr-90</b>	<b>8.00E-10</b>	<b>1.20E-12</b>
<b>Th-232</b>	<b>1.00E-13</b>	<b>1.20E-15</b>
<b>U-235</b>	<b>6.00E-11</b>	<b>6.00E-13</b>

## **Attachment 3**

# **Work Instruction and Standard Operating Procedures**



## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

## 1.0 SAMPLING FREQUENCY

Total suspended particulate (TSP)/particulate matter smaller than 10 microns in diameter (PM10) high-volume air samplers are operated daily, concurrent with field activities that result in earth movement through excavation or transport of materials that lead to dust generation. Although the sampling frequency may vary, sampling duration is limited to daily fieldwork duration.

## 2.0 BACKGROUND

This document outlines procedures for air sampling for TSP and PM10 using high-volume air samplers. The procedures are consistent with the sampling methods described in Title 40 Code of Federal Regulations, Part 50, Appendix B for TSP and Title 40 Code of Federal Regulations, Part 50, Appendix J for PM10.

## 3.0 TOOLS, EQUIPMENT, AND MATERIALS

- One clean (white), undamaged, pre-weighed (tared) U.S. Environmental Protection Agency-approved glass-fiber or quartz filter protected by glassine envelope. Ensure the filter is unblemished and unfolded. (A pre-folded filter hinders accurate sample collection by weakening the web fiber structure at the crease and allowing the smallest particles to pass through. Additionally, a folded filter causes the manometer readings [air flow through the filter paper] to be inaccurate.)
- Clean, disposable gloves (powder free) for handling the filters.
- One filter cassette assembly (Figure 1).
- One 9-inch by 12-inch particulate sampler field envelope.
- A logbook and ballpoint pen, to record data taken. **A pencil should never be used**, as the lead can smear or rub off during usage. If a mistake is made, draw a single line through the incorrect data in ink, add the correct data in ink, and initial the change near the correction.
- A bag with a shoulder strap to carry the items listed above.

## 4.0 EXPOSURE LIMITS

Exposure limits are provided in project-specific planning documents.

## 5.0 PREPARATORY PROCEDURE FOR FILTER CHANGES

1. Before setting up the samples, clean and dry the working surface of dust or other contaminants.
2. Open the filter cassette by removing the protective cover and unscrewing the thumbscrew, which secure the top of the frame to the screen support (Figure 1).
3. After cleaning hands and donning a pair of clean, disposable gloves, then select a clean sample filter. Each filter will be marked with an ID number located on the back of the filter in





## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

the upper right corner. Inspect the filter for pinholes, tears, abrasions, loose material, discoloration, and other non-uniformities. Discard defective or damaged filters.

4. On the "Air Monitoring Sample Collection Log" (Appendix A), record and make note of the filter ID number and run date.
5. Place the filter into an open filter cassette centering the filter over the support screen. Ensure that the filter ID number is facing down. (The upward or exposed side of the filter has a textured surface.) Close the filter cassette by replacing the top of the frame over the filter ensuring that the gasket material covers the outer edges of the filter. Tighten the thumbscrews until the gasket material seal firmly on the filter and bottom support. (Note: The seal should be firm, but do not over-tighten the thumbscrews, this may damage the filter material.) Replace the filter cassette cover. The filter cassette cover must be in place to protect the filter during transport to and from the sample site.

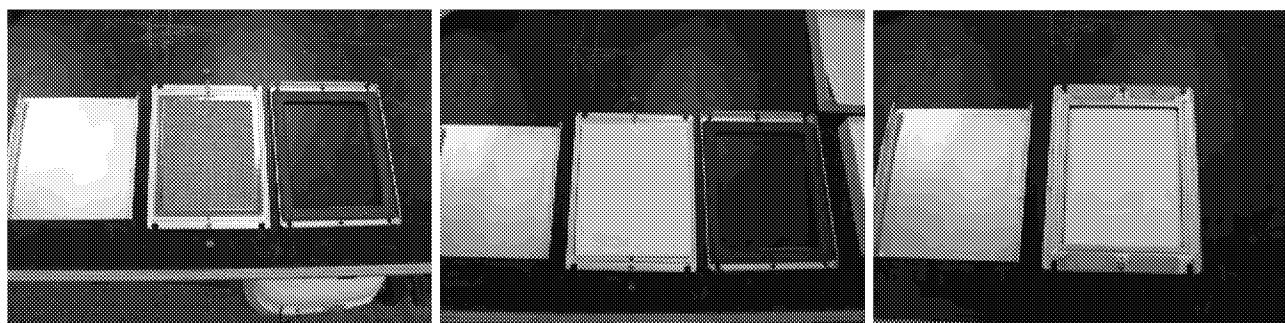


Figure 1: Filter Cassette Loading

## 6.0 ON-SITE INSTALLATION AND SETUP FOR A SAMPLE RUN

Upon arrival at the monitoring site, unlock the security fencing and inspect the equipment (in the comment section on the "Air Monitoring Sample Collection Log" [Appendix A], note items needing maintenance or irregularities that may affect data quality):

1. Open the main door to sampler; ensure that the sampler is turned off.
2. Check that the generator gas level is full, and the generator can run nonstop for the duration of the sampling period (8 to 10 hours for a workday, 24-hours, or other as specified in project-specific plans). Make a note of adjustments on the "Air Monitoring Sample Collection Log."
3. Unlatch and open the top of the sampler.
4. If a filter is present, remove the old filter cassette by unscrewing the thumb screws until the studs can be pushed out of the notches and away from the filter cassette.
5. Install the new filter cassette.
  - a. Slide studs into notches and tighten thumb screws.
  - b. Tighten the nuts evenly until they are snug.
  - c. Do not over-tighten.



## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

6. Remove the aluminum metal snap cover from the new filter cassette and use it to cover the used filter cassette.
7. Close and latch the top of the sampler.
8. Turn on the generator. Let the generator stabilize (approximately five minutes).
9. Manually turn on the sampler.
10. After the flow controller stabilizes (approximately five minutes), check the flow meter reading and record the current time on the "Air Monitoring Sample Collection Log."
11. Close the doors to the sampler and lock security fence (if present).

## 7.0 SAMPLE RECOVERY PROCEDURE FOR POST SAMPLING

Upon return to the work area and before recovering the samples, ensure the area is clean and dust free.

1. Review the information recorded in the "Air Monitoring Sample Collection Log" (Appendix A). Confirm the information recorded is consistent and correct.
2. Gather the necessary materials to recover and store the samples (clean, disposable gloves, protective paper sheets [used to separate the clean filters], and envelopes).
3. Clean hands and don clean, disposable gloves.
4. Unlatch and open the top of the sampler.
5. Manually turn off the sampler. Record the time in the "Air Monitoring Sample Collection Log."
6. Turn off the generator.
7. Remove the filter cassette by unscrewing the thumb screws until the studs can be pushed out of the notches and away from the filter cassette. If weather allows, open the filter cassette by removing the thumb screws and remove the top of the filter cassette. **Care must be taken to not lose particulate matter from the filter.**
8. Remove the filter from the filter cassette and carefully fold the filter lengthwise so the exposed (collected sample) side is folded inward onto itself to prevent loss of particulate matter. The edges of the filter should be even. (Take care not to bend or tear the corners.) The field ID number will be on the outside of the folded filter.
9. Make sure the filter ID number on the filter envelope and "Air Monitoring Sample Collection Log" matches the filter number on the filter.
10. Place the filter into the protective envelope.
11. Close the envelope by tucking the flap inside the lip of the envelope. (Do not lick and seal the adhesive.)
12. Label the exterior of the envelope with the site name, sampler ID, filter ID number, and sample run date.



## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

13. Record sample information on the chain of custody (COC) form and package the samples for shipment to the off-site analytical lab.

## 8.0 SAMPLE HANDLING AND SHIPPING PROCEDURES

The field chemist will prepare and ship the filters collected for the workweek to the off-site analytical lab for processing and chemical analysis.

1. Inventory the filters by comparing the filter ID numbers and sample run dates shown on the envelopes with the information record in the "Air Monitoring Sample Collection Log" (Appendix A) to make sure the samples are accounted for and ready for shipment.
2. Verify filter sample information on the "Air Monitoring Sample Collection Log" to the COC forms and labels and package samples for submittal to the analytical lab. Ship samples in a rigid container with sufficient packing material to prevent jostling or damage to the sample filters.
3. A copy of the COC form will be retained in the sample's shipment monitoring file.
4. The filter samples and COC forms will be shipped via courier through the U.S. Postal Service.



## Hunters Point Naval Shipyard Procedure

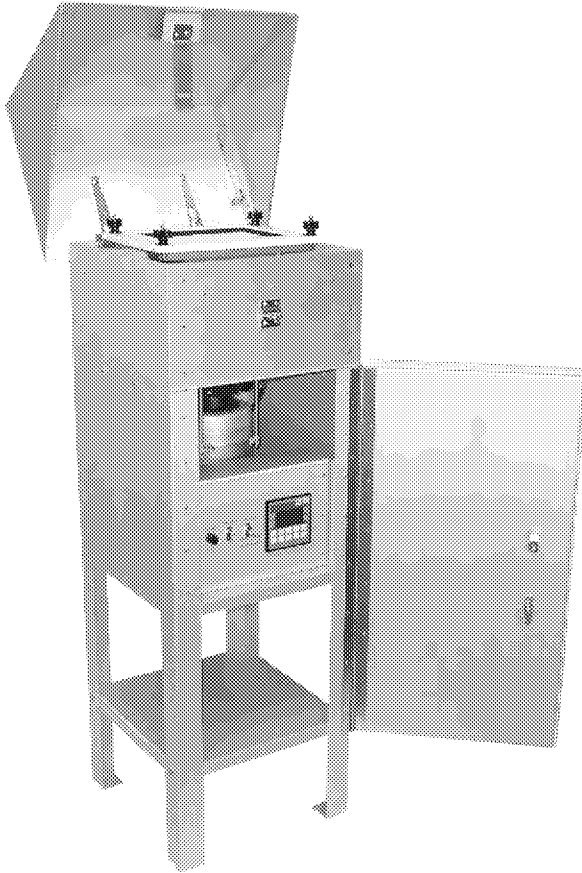
Revision: 1

Approval Date: 4/23/2020

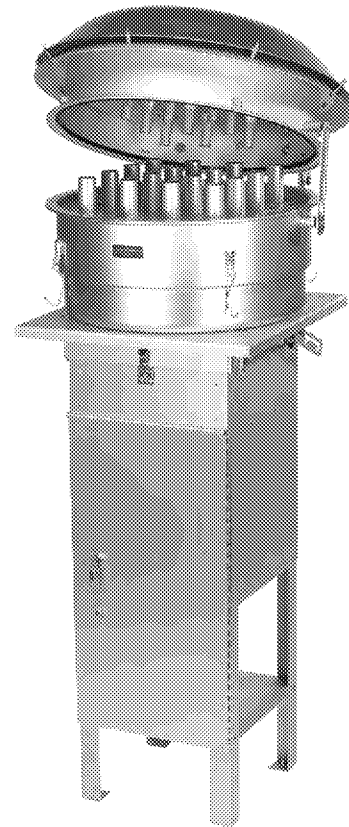
# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

## 9.0 INSTRUMENT PARTS

The sampling procedures described pertain to both the PM10 and TSP samplers. The following instrument details are shown for user reference:



**Hi-Vol TSP Sampler (Typical)**



**Hi-Vol PM10 Sampler (Typical)**



## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

### Automatic Flow Control Brushless Blower

Motor Mounting Plate

Mass Flow Control System

Graphic LCD Display Panel

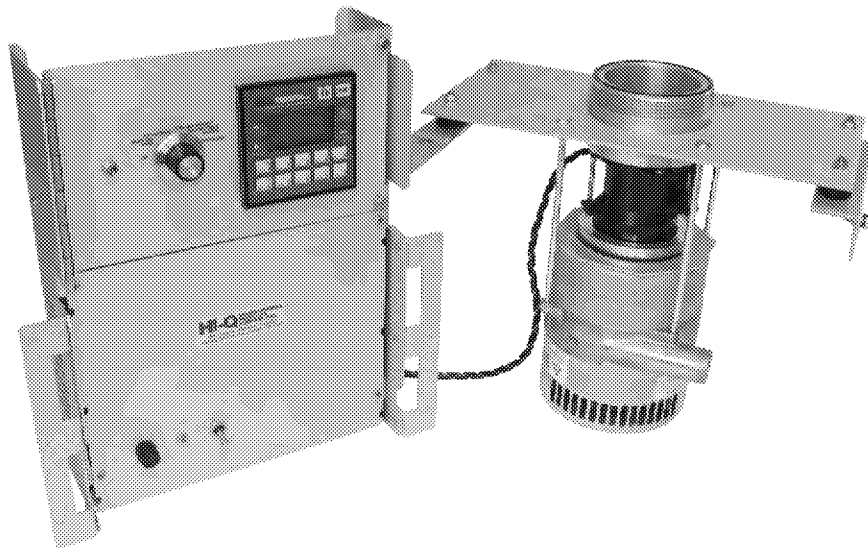
Power Toggle Switch

Fuse Holder/Fuse

Blower Exhaust

Motor Speed Controller

Brushless Blower





## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

### Manual Speed Control Brushless Blower

Motor Mounting Plate

Electronic Elapsed Resettable Timer

Motor Speed Controller

Power Toggle Switch

Fuse Holder/Fuse

Blower Exhaust

Brushless Blower





## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

### Filter Holder and Filter Cassette Assembly

Plastic Thumb Nuts

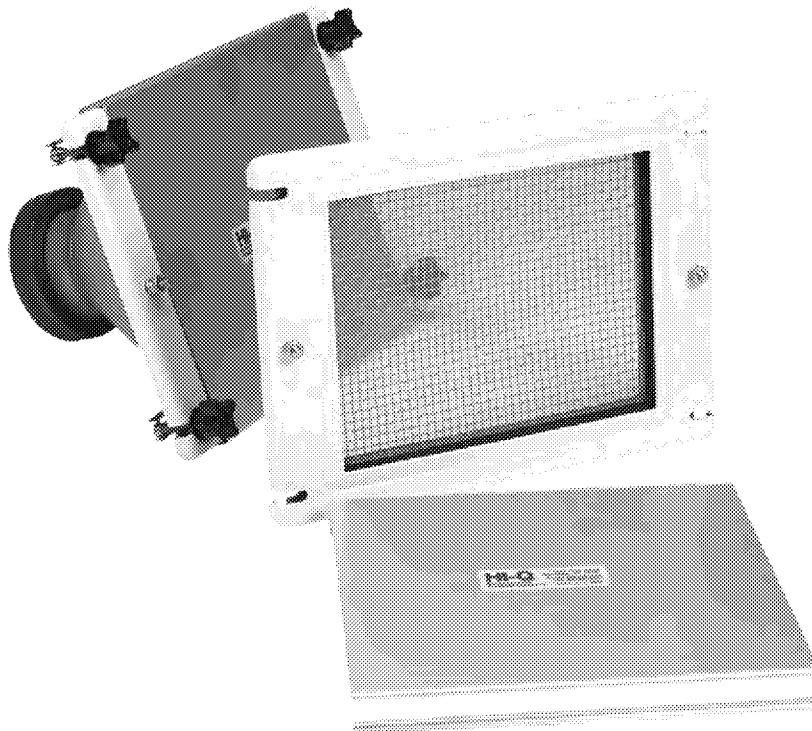
Filter Holder

Aluminum Hold Down Frame

Aluminum Filler with Stainless Steel Screen

Brass Thumb Nuts

Aluminum Snap-On Cover





## Hunters Point Naval Shipyard Procedure

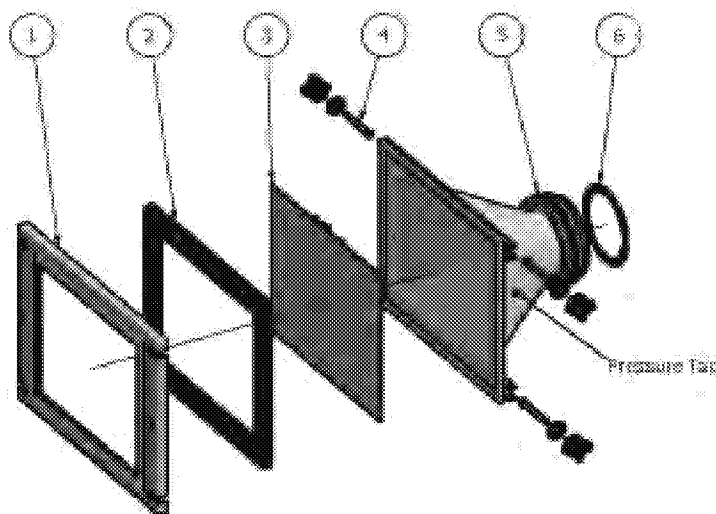
Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

### Filter Holder Assembly

1. Hold Down Frame
2. 8-inch by 10-inch Gasket
3. Filter Paper
4. Plastic Thumb Nut, Brass Bolt, Washer, and Rivet
5. Aluminum Threading Ring
6. Filter Holder Gasket (between filter holder and blower motor)







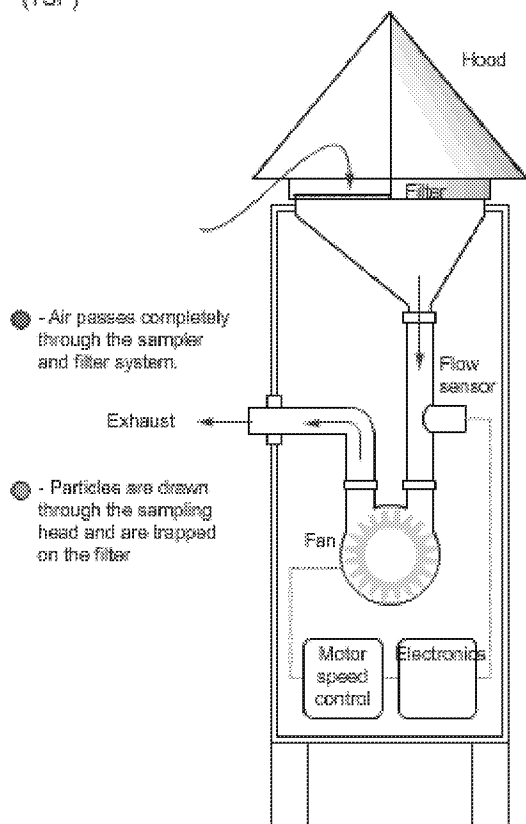
# Hunters Point Naval Shipyard Procedure

Revision: 1

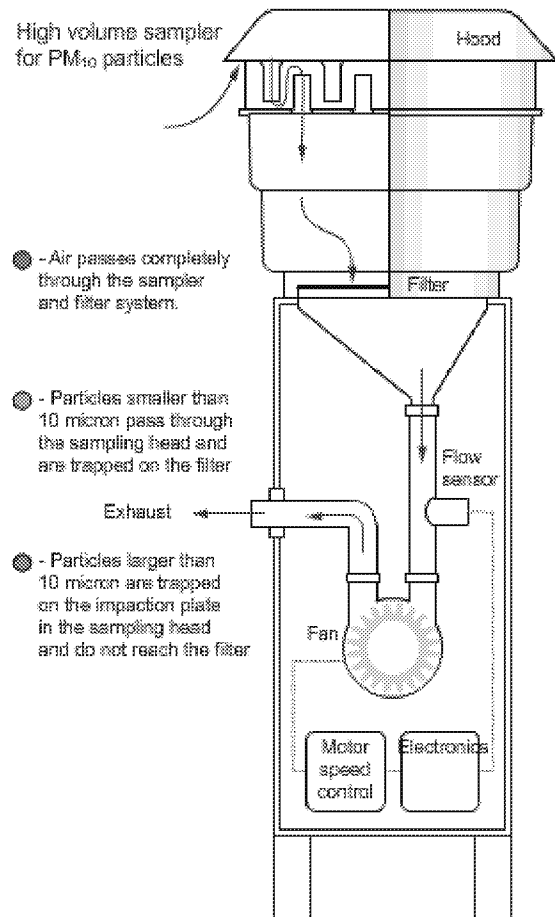
Approval Date: 4/23/2020

## HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

High volume sampler  
for Total Suspended Particulates  
(TSP)



High volume sampler  
for PM<sub>10</sub> particles





# Hunters Point Naval Shipyard Procedure

Revision: 1

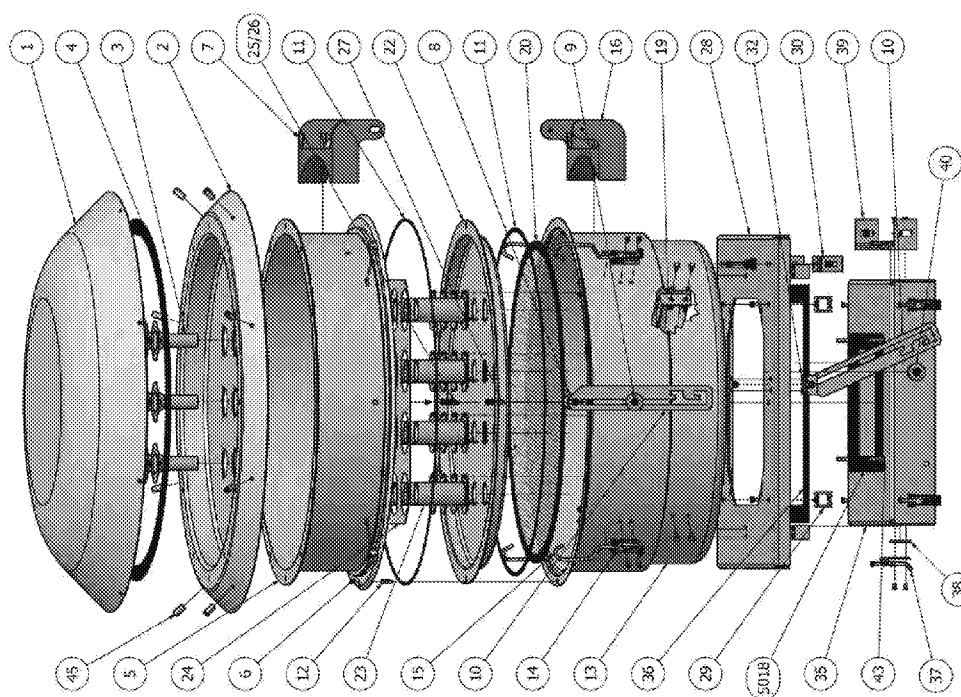
Approval Date: 4/23/2020

## HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

### PM10 Size Selective Inlet Component Parts

1. Hood
2. Acceleration Nozzle Plate w/ Nozzles
3. Acceleration Nozzle, each
4. Acceleration Nozzle Gasket
5. Top Tub Housing
6. Top Tub Housing Strike
7. Top Tub Housing Hinge
8. Tub Housing Large Strut Holder
9. Shoulder Bolt, Small
10. Strut
11. Bead Gasket Strip (Between Tubs)
12. Brass Alignment Pin, Large
13. Bottom Tub Housing
14. Bottom Tub Housing Catch (No Hook)
15. Bottom Tub Housing Catch Hook, each
16. Bottom Tub Housing Hinge
19. Bug Screen Support Angle, each
20. Bug Screen w/ Safety Edging
21. Bug Screen Safety Edging Only
22. Second Stage Plate w/ 16 Vent Tubes

23. Vent Tube (16 Required)
24. Shim Plate
25. Shim Clip (2 Required)
26. Spring for Shim Clip (2 Required)
27. Small Brass Alignment Pin, each
28. Inlet Base Pan
29. Inlet Base Pan Strike (6 Required)
30. Inlet Base Pan Hinge Bracket (2 Required)
31. Inlet Base Pan Hinge Shoulder Bolt, each
32. Inlet Base Pan Strut Bracket
35. Shelter Base Pan
36. Shelter Base Pan Gasket 16" X 16"
37. Shelter Base Pan Catch w/ Bolt (6) each
38. Shelter Base Pan Catch Spacer (6) each
39. Shelter Base Pan Hinge Bracket (2) each
40. Shelter Base Pan Strut Holder Space, Small
43. Brass Bolt Assembly w/ Nuts for FH
44. Hood Spacers (8 Required), each
45. Hood Spacers (Bag of 8 w/ Hardware)
5018. Shelter Base Pan Filter Holder Gasket





## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

## 10.0 REFERENCES

Code of Federal Regulation, Title 40, Part 50, Appendix B, "Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High-Volume Method)."

Code of Federal Regulation, Title 40, Part 50, Appendix, "Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere."

Code of Federal Regulation, Title 40, Part 53, Subpart D, "Procedures for Testing Performance Characteristics of Methods for PM10."

U.S. Environmental Protection Agency, 1990, *Quality Assurance Guidance Document*, Section 2.11, "Monitoring PM10 in Ambient Air using a High-Volume Sampler Method."

U.S. Environmental Protection Agency, 2004, *Air Quality Criteria for Particulate Matter (Final Report)*, EPA 600/P-99/002aF-bF.

U.S. Environmental Protection Agency, 2007, *Guidance for the Preparation of Operating Procedures for Quality Related Operations*, EPA QA/G6.

U.S. Environmental Protection Agency, 2017, *Quality Assurance Handbook for Air Pollution Measurement Systems*.

## 11.0 EXHIBITS

Figure 1	Filter Cassette Loading
Appendix A	Air Monitoring Sample Collection Log
Appendix B	Chain of Custody



Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

## APPENDIX A: AIR MONITORING SAMPLE COLLECTION LOG



DATE		20
PAGE:	OF	

### AIR MONITORING SAMPLE COLLECTION LOG

PROJECT NAME **CTO 0024 HPNS - Parcel E Remedial Action - Phase 1**

PROJECT No. **500712**

SAMPLE TYPE **Air (Grab)**

ANALYSES	CONTAINER	ANALYSES	CONTAINER
<input checked="" type="checkbox"/> TSP	<input checked="" type="checkbox"/> 6x10 Whatman QM-A Quartz Filter	<input type="checkbox"/> PCBs (EPA method TO-04)	<input type="checkbox"/> Polyurethane foam (PUF) trap
<input checked="" type="checkbox"/> PM-10	<input checked="" type="checkbox"/> 8x10 Whatman QM-A Quartz Filter	<input type="checkbox"/> PAHs (EPA 8270-SIM TO-13)	<input type="checkbox"/> Polyurethane foam (PUF) trap
<input checked="" type="checkbox"/> Asbestos (NIOSH 7400)	<input checked="" type="checkbox"/> 0.8µ PCM filter	<input type="checkbox"/> Dioxin (EPA method TO-9A)	<input type="checkbox"/> Polyurethane foam (PUF) trap

#### Field Conditions:

☐ Sunny    ☐ Overcast    ☐ Rain    ☒ Wind    ☒ Temperature  
Precipitation: \_\_\_\_\_ in.    Wind Gust: \_\_\_\_\_ mph    Average Temp: \_\_\_\_\_ °F    Pressure: \_\_\_\_\_ in.

Runtime				Analysis	Station Code		Sample ID		Comments:
Start		Finish					Site-AnalysisDate-StationCodeLocation		
Date	Time	Date	Time				PE-TSP110119-B606UPWIND		
				TSP				PE-TSP_____B606UPWIND	
				PM-10				PE-PM10_____B606UPWIND	
				Asbestos				PE-ASBESTOS_____B606UPWIND	

Runtime				Analysis	Station Code		Sample ID	Comments:
Start		Finish			B606			
Date	Time	Date	Time					
				TSP			PE-TSP.....-B606DOWNWIND	
				PM-10			PE-PM10.....-B606DOWNWIND	
				Asbestos			PE-ASBESTOS.....-B606DOWNWIND	

- All samples analyzed by Eurofins Calscience, LLC

The monitoring stations will be supplemented with real-time PM10 air monitoring upwind and downwind of Building 606.

PREPARED BY: \_\_\_\_\_



## Hunters Point Naval Shipyard Procedure

Revision: 1

**Approval Date: 4/23/2020**

## HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM<sub>10</sub>

## APPENDIX B: CHAIN OF CUSTODY



APTIS Federal Services, LLC  
4605 Park Chicago Hwy  
Concord, CA 94520

## CHAIN OF CUSTODY

Ref. Document # ETOL - AIR 150  
Page 1 of 4

Project Manager: Nels Johnson

Send Report To: *Eddie Kalombo*

Phone/Fax Number: 415.987.0760

Address: 4005 Port Chicago Hwy

City: Concord, CA 94520

[edite.kalombo@april.fr](mailto:edite.kalombo@april.fr)

0: KKK

Project Number: 500506

Project Name: HPNS - Parcel E-2

Project Location: San Francisco

Purchase Order #: 202935

Delivery Date: 8/6/2018

Waybill Number: N/A

Lab Destination: Coscience

7440 Lincoln Way

Garden Grove CA 92841

**Lab Contact:** Terri Chang

Surgeon's Name(s): KKK

### Citation Information

Contains Type

Analysis Requested									
PCB (EPA 5082) / TOC	PAH (EPA 8270-SEM) / TOX	Asbestos (NIOSH 7400)	PM10-40 (EPA Subpart BAAQMD Reg 6)	TSP, M <sub>10</sub> , PM <sub>10</sub> (40 CFR 50.8 App B, NIOSH 7500-60)	Flow Rate (L/min.)	Sample Volume (m <sup>3</sup> )			
				X	1132.8	1624.44			
				X	1132.8	1624.44			
			X		1132.8	1624.44			
			X		1132.80	1624.44			
		X			2.00	2.868			
		X			2.00	2.868			
				X	1132.80	1632.36			
				X	1132.80	1627.83			

[illegible]

Special Instructions:	
-----------------------	--

Turn Around Time				Level Of QC Required:		Method Codes	
<input type="checkbox"/> 24-hr <input type="checkbox"/> 5-day <input type="checkbox"/> 18-day		I	II	III	Project Specific:	C = Composite	U = Split
						Matrix Codes	
Reinspected By:	Date:	Time:	Reinspected By:	Date:	Time:	CPH = Enslaving Water	SO = Soil
						CW = Cleaned Water	SL = Sludge
Reinspected By:	Date:	Time:	Reinspected By:	Date:	Time:	WW = Waste Water	CS = Chip Samples
						S = Air	AS = Air, Gasoline, PFAS Free Gasoline



# Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

## HIGH-VOLUME AIR SAMPLING PROCEDURES FOR TSP/PM10

### APPENDIX B: CHAIN OF CUSTODY (CONTINUED)

AIR MONITORING LOG										
PROJECT NAME: HPNS Parcel E-2				PROJ. NO. 506506		Asbestos	TSP	PAH	PCB	
STATION						COC# 001				
SAMPLE NO. HP-ST13-PCB120716-UPWIND						12/7/16 Station 13 Upwind				
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CEL-008	34.0	34.0	34.0	12/07/16 07:40	12/08/16 08:15	1475	1404.2	PCB	962.85	
SAMPLE NO. HP-ST14-PCB120716-DOWNWIND						12/7/16 Station 14 Downwind				
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CEL-074	34.0	34.0	34.0	12/07/16 07:50	12/08/16 08:30	1480	1408.0	PCB	962.85	
SAMPLE NO. HP-ST13-PAH120716-UPWIND						12/7/16 Station 13 Upwind				
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CEL-063 B	34.0	34.0	34.0	12/07/16 07:40	12/08/16 08:15	1475	1404.2	PAH	962.85	
SAMPLE NO. HP-ST14-PAH120716-DOWNWIND						12/7/16 Station 14 Downwind				
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CEL-082 T	34.0	34.0	34.0	12/07/16 07:50	12/08/16 08:30	1480	1408.0	PAH	962.85	
SAMPLE NO. HP-ST13-ASBESTOS120716-UPWIND						12/7/16 Station 13 Upwind				
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CO701766	2.0	2.0	2.0	12/07/16 07:40	12/08/16 08:15	1475	2.9	Asbestos	56.64	
SAMPLE NO. HP-ST14-ASBESTOS120716-DOWNWIND						12/7/16 Station 14 Downwind				
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CO701678	2.0	2.0	2.0	12/07/16 07:50	12/08/16 08:30	1480	3.0	Asbestos	56.64	
SAMPLE NO. HP-ST13-TSP120716-UPWIND						12/7/16 Station 13 Upwind				
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
Q0364107	40.0	40.0	40.0	12/07/16 07:40	12/08/16 08:15	1475	1652.0	TSP	1132.80	
SAMPLE NO. HP-ST14-TSP120716-DOWNWIND						12/7/16 Station 14 Downwind				
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
Q0364108	40.0	40.0	40.0	12/07/16 07:50	12/08/16 08:30	1480	1657.6	TSP	1132.80	



## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

## 1.0 SAMPLING FREQUENCY

Asbestos air samplers are operated daily, concurrent with field activities resulting in earth movement through excavation or transport of materials that lead to dust generation. Although the sampling frequency may vary, sampling duration is limited to daily fieldwork duration.

## 2.0 BACKGROUND

This document outlines air sampling procedures for asbestos comparison with the occupational exposure limits (OELs) in the Occupational Safety and Health Administration (OSHA) and American Conference of Governmental Industrial Hygienists threshold limit values. This document does not cover sampling for U.S. Army or U.S. Environmental Protection Agency asbestos environmental or clearance levels. There are two methods used to sample and analyze for asbestos occupational exposure. Both methods use the same basic sampling procedures; however, they use different techniques for analyzing asbestos. The first method uses phased contrast microscopy (PCM) and the second method uses transmission electron microscopy (TEM).

## 3.0 TOOLS, EQUIPMENT, AND MATERIALS

- Two clean, undamaged, pre-weighed (tared) U.S. Environmental Protection Agency-approved 25-millimeter (mm) mixed cellulose ester (MCE) filter cassettes with 50-mm cowl (Figure 1).
- Two Gilian GilAir® Plus sampling pumps (or equivalent).
- Clean, disposable gloves (powder free) for handling filters.
- A logbook and ballpoint pen to record data taken. **A pencil should never be used**, as the lead can smear or rub off during usage. If a mistake is made, draw a single line through the incorrect data in ink, add the correct data in ink, and initial the change near the correction.
- A bag with a shoulder strap to carry the items listed above.

## 4.0 EXPOSURE LIMITS

There are two OELs for asbestos:

- Eight-hour time-weighted average, threshold limit value and the OSHA permissible exposure limit of 0.1 fibers per cubic centimeter of air.
- OSHA excursion limit of 1 fiber per cubic centimeter of air for 30 minutes. (To collect a sample for comparison to the OSHA excursion limit, it is recommended to only sample for 30 minutes. It is also recommended to collect multiple samples during the expected period of the highest concentration of asbestos fibers.)

## 5.0 PHASED CONTRAST MICROSCOPY METHOD (HUNTERS POINT NAVAL SHIPYARD METHOD)

The PCM method is the most common asbestos sampling method available to determine worker compliance with the OEL. However, the PCM method does not distinguish between asbestos and

Page 1 of 9

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## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

non-asbestos fibers. The PCM method's analysis is a visual counting method; therefore, it is imperative that the filter be free of dust.

- The PCM method used at Hunters Point Naval Shipyard is based on the National Institute for Occupational Safety and Health 7400 Method (1994) and has been modified by both the U.S. Army in U.S. Public Health Command (Provisional) *Technical Guide 141*, "Industrial Hygiene Sampling Guide," and OSHA in their general industry and construction standards (Title 29 Code of Federal Regulations Part 1910.1001 and Part 1926.1101).
- The PCM method requires the use of a 25-mm MCE filter cassette in an electrically-conductive-plastic filter-holder with a 50-mm cowling (Figure 1).
- The filter cassette is placed in the employee's breathing zone or in the area at the breathing-zone height (between 4 to 6 feet from the ground).
- To sample, remove the inlet cover from the filter cassette (or face cover of inlet cover)—this is called "sampling open face"—and connect the suction side of the filter cassette to the pump, then place the filter cassette at the location intended to sample with the face pointed down (the face of the cassette will point to the ground).
- If the 25-mm filter cassette is used, then the method recommends collecting a minimum of 400 liters of air from between 0.5 and 16 liters per minute (L/min); however, a flow rate between 2 and 4 L/min is recommend.
- Because the PCM method analysis is adversely affected by accumulations of dust on the filter cassette, monitor the filter condition, and if a buildup of dust is noted on the filter, then the filter cassette should be changed and the times recorded. Both filters will be submitted for laboratory analysis.
- If 25-mm filters are not available, then, as an alternate, use a 37-mm filter cassette with MCE filter; however, if a 37-mm filter cassette is used, collect a larger minimum volume of approximately 3,000 liters of air at 2 to 10 L/min.
- In addition to the samples, 2 field blanks should be included with the first 10 samples, plus 1 additional blank for every additional 10 samples.
- Ship samples in a rigid container with sufficient packing material to prevent jostling or damage to the filter cassettes.

## 6.0 TRANSMISSION METHOD MICROSCOPY

The TEM method is the least common asbestos sampling method available to determine worker compliance with the OEL. Unlike the PCM method, the TEM method identifies and counts only asbestos fibers. The TEM method's analysis uses an electron microscope; therefore, it can better distinguish between asbestos and non-asbestos fibers (the removal of dust from the filter media is not as imperative as it is with the PCM method).

- The TEM method is based on the National Institute for Occupational Safety and Health 7402 Method (1994) and has been modified by both the U.S. Army in U.S. Public Health Command (Provisional) *Technical Guide 141*, "Industrial Hygiene Sampling Guide," and OSHA in their

Page 2 of 9

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## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

general industry and construction standards (Title 29 Code of Federal Regulations Part 1910.1001 and Part 1926.1101).

- The TEM method requires the use of a 25-mm MCE or polycarbonate filter in an electrically-conductive-plastic filter-holder, with a 50-mm cowling (Figure 1). The filter cassette is handled in the same manner as the PCM method (Section 5.0) and the same flow rates and volumes are collected.

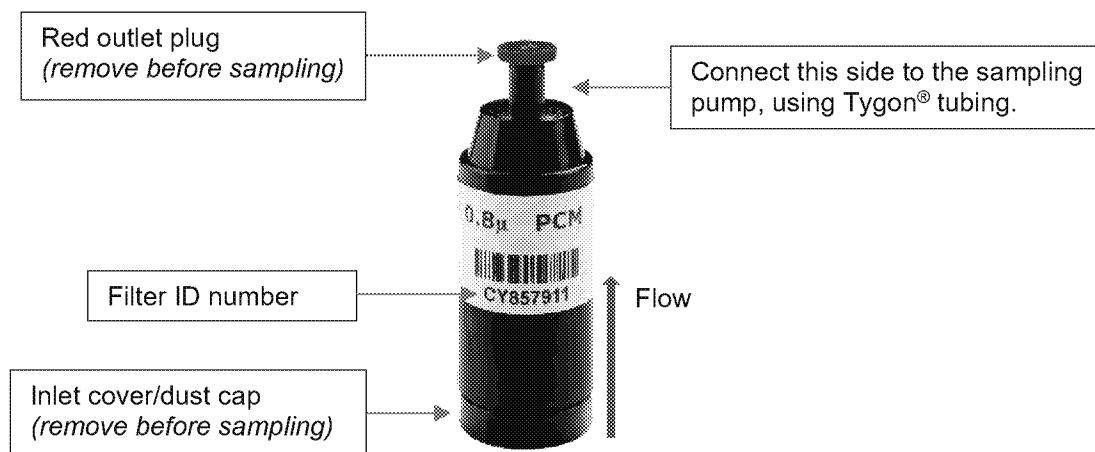


Figure 1: Recommended 25-mm Mixed Cellulose Ester Filter Cassette with 50-mm Cowling

## 7.0 ASBESTOS PRE-SAMPLING ACTIVITIES

- Procure two Gilian GilAir® Plus units (or equivalent) from manufacturer or supplier.
- Assemble the pump in accordance with the user manual.
- Calibrate the pump to the desired flow rate of 2 L/min.
- Charge the pumps and batteries that will be used during the sampling.
- Procure sample filters from the subcontract laboratory.

## 8.0 ASBESTOS SAMPLING PROCEDURE

Upon arrival at the monitoring site, unlock the security fencing and inspect the equipment (in the comment section on the "Air Monitoring Sample Collection Log" [Appendix A], note items needing maintenance or irregularities that may affect data quality):

- After cleaning hands and donning a clean pair of disposable gloves, then select a clean sample filter cassette and inspect the filter cassette for pinholes, tears, abrasions, loose material, discoloration, and other non-uniformities. Discard defective or damaged filters.
- Remove the inlet cover from the MCE filter cassette. Attach the MCE filter cassette to the pump via tubing.



## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

3. Record the following information on the "Air Monitoring Sample Collection Log" (Appendix A):
  - a. Date and time of sampler setup visit
  - b. Site identification and location
  - c. Sampler model, unique sample ID number (this is the filter ID number—as printed on each filter)
  - d. Unusual conditions that may affect samples (e.g., subjective evaluation of pollution on the that day, construction activity, weather conditions)
  - e. Setup operator's signature or initials
4. Press and hold the POWER/ENTER key until the pump displays the **Startup Screen**.
5. Place the pump, tubing, and filter cassette in the desired sampling location.
6. Use the navigation keys (up/down arrows) to select **Run**.
7. Press the POWER/ENTER key to begin sampling. Note: Before the pump enters the **Run** mode, the pump will go into a self-calibration mode for 7 to 10 seconds. During this interval, "Sensor Calibration" will display on the screen.
8. After the flow controller stabilizes (approximately one minute), check the flow meter reading.
9. Record the current date and time on the "Air Monitoring Sample Collection Log."
10. Close the doors to the sampler and lock security fence (if present).

## 9.0 ASBESTOS SAMPLE RECOVERY PROCEDURE FOR POST SAMPLING

Upon return to the work area and before recovering the samples, ensure the area is clean and dust free:

1. Review the information recorded in the "Air Monitoring Sample Collection Log" (Appendix A). Confirm the information recorded is consistent and correct.
2. Gather the necessary materials to recover and store the samples (clean disposable gloves, MCE filter cassette inlet cover, resealable-plastic storage bag).
3. Clean hands and don clean, disposable gloves.
4. From any display on the pump, press POWER/ENTER. The **Pause/Stop Menu** will appear in the upper left corner of the display.
5. Select Stop and press POWER/ENTER to stop the sample.
6. When pump is not running or in a program pause, press and hold POWER/ENTER key. Continue to hold POWER/ENTER until power down window appears and countdown completes in order to manually turn the sampler off.
7. Record the following information on the "Air Monitoring Sample Collection Log":
  - a. Stop time and total elapsed time of the sample run
  - b. Average flow rate, coefficient of variation of the flow rate, and total volume sampled

Page 4 of 9

PRINTED BLANK FORM IS AN UNCONTROLLED COPY, FORM WITH DATA ENTERED IS CONTROLLED  
IN ACCORDANCE WITH PROJECT/SITE DOCUMENT MANAGEMENT REQUIREMENTS



## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

- c. Conditions at the site or of the collector that may have affected the sample
  - d. Flags triggered by the sampler (e.g., power outage, flow rate variation)
  - e. Explanations for questionable or voided samples
  - f. Collecting operator's signature or initials
8. Place the inlet cover on the MCE filter cassette and place the filter into the protective resealable bag.
  9. Label, record sample information on the chain of custody (COC) (Appendix B), and package the samples for shipment to the off-site analytical lab.

## 10.0 SAMPLE HANDLING AND SHIPPING PROCEDURES

The field chemist will prepare and ship the filters collected for the workweek to the off-site analytical lab for processing and analysis:

1. Inventory the filters by comparing the filter ID numbers and sample run dates shown on the information recorded in the "Air Monitoring Sample Collection Log" (Appendix A) to ensure samples are accounted for and ready for shipment.
2. Verify filter sample information on the "Air Monitoring Sample Collection Log" to the COC forms (Appendix B) and labels and package samples for submittal to the analytical lab. Ship samples in a rigid container with sufficient packing material to prevent jostling or damage to the filter cassettes.
3. A copy of the COC form will be retained in the sample's shipment monitoring file.
4. The filter samples and COC forms will be shipped via laboratory courier.



## Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

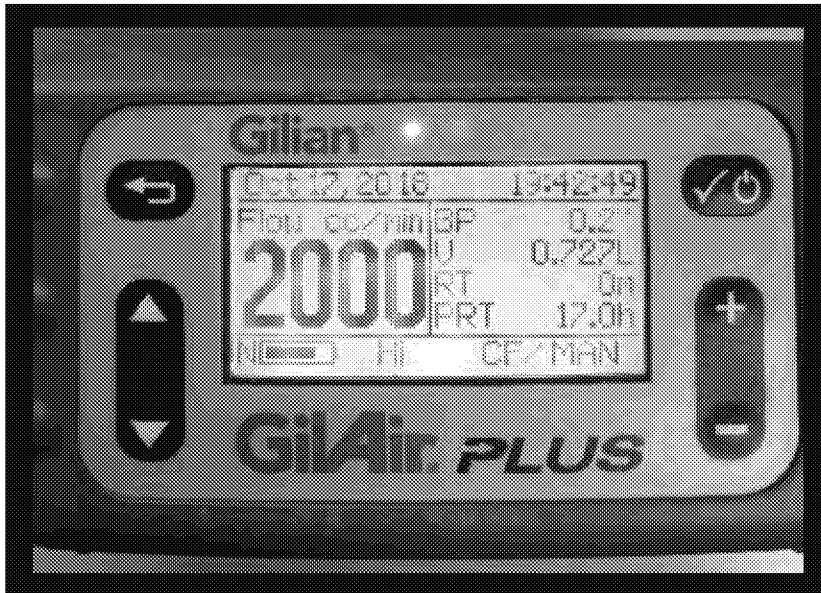


Figure 2: Gilian GilAir® Plus Sample Run Display

## 11.0 REFERENCES

American Conference of Governmental Industrial Hygienists, 2009, *Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices*.

National Institute for Occupational Safety and Health, 1994, *Manual of Analytical Methods*, 4th Edition, Publication No. 94-113, 1994.

Occupational Safety and Health Administration, *Asbestos Standard*, Title 29 Code of Federal Regulations Part 1910.1001 and Part 1926.1101, most current edition.

U.S. Environmental Protection Agency, 1987, *Asbestos Hazard Emergency Response Act*, "Modified Transmission Electron Microscopy," Title 40 Code of Federal Regulations part 763 Appendix A Subpart E.

U.S. Public Health Command (Provisional), 2010, *Technical Guide 141*, "Industrial Hygiene Sampling Guide," October.

## 12.0 EXHIBITS

Figure 1	Recommended 25-mm Mixed Cellulose Ester Filter Cassette with 50-mm Cowling
Figure 2	Gilian GilAir® Plus Sample Run Display
Appendix A	Air Monitoring Sample Collection Log
Appendix B	Chain of Custody



# Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

## LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

### APPENDIX A: AIR MONITORING SAMPLE COLLECTION LOG



DATE		20
PAGE:	OF	

#### AIR MONITORING SAMPLE COLLECTION LOG

PROJECT NAME CTO 0024 HPNS - Parcel E Remedial Action - Phase 1

PROJECT No. 500712

SAMPLE TYPE Air (Grab)

ANALYSES	CONTAINER	ANALYSES	CONTAINER
<input checked="" type="checkbox"/> TSP	<input checked="" type="checkbox"/> 8x10 Whatman QM-A Quartz Filter	<input type="checkbox"/> PCBs (EPA method TO-04)	<input type="checkbox"/> Polyurethane foam (PUF) trap
<input checked="" type="checkbox"/> PM-10	<input checked="" type="checkbox"/> 8x10 Whatman QM-A Quartz Filter	<input type="checkbox"/> PAHs (EPA 8270-SM TO-13)	<input type="checkbox"/> Polyurethane foam (PUF) trap
<input checked="" type="checkbox"/> Asbestos (NIOSH 7400)	<input checked="" type="checkbox"/> 0.6µ PCM filter	<input type="checkbox"/> Dioxin (EPA method TO-9A)	<input type="checkbox"/> Polyurethane foam (PUF) trap

<b>Field Conditions:</b> <input type="checkbox"/> Sunny <input type="checkbox"/> Overcast <input type="checkbox"/> Rain <input checked="" type="checkbox"/> Wind <input checked="" type="checkbox"/> Temperature Precipitation: _____ in.    Wind Gust: _____ mph    Average Temp: _____ °F    Pressure: _____ in.									
Runtime				Analysis	Station Code	Sample ID	Comments		
Start		Finish							
Date	Time	Date	Time		B606	Site Analysis Date-Station Code-Location			
				TSP		PE-TSP110119-B606UPWIND			
				PM-10		PE-PM10_____-B606UPWIND			
				Asbestos		PE-ASBESTOS_____-B606UPWIND			
Runtime				Analysis	Station Code	Sample ID	Comments		
Start		Finish							
Date	Time	Date	Time		B606				
				TSP		PE-TSP_____-B606DOWNWIND			
				PM-10		PE-PM10_____-B606DOWNWIND			
				Asbestos		PE-ASBESTOS_____-B606DOWNWIND			
- All samples analyzed by Eurofins Calscience, LLC The monitoring stations will be supplemented with real-time PM10 air monitoring upwind and downwind of Building 606.									

PREPARED BY: \_\_\_\_\_



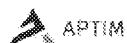
# Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

## LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

### APPENDIX B: CHAIN OF CUSTODY



APTIM Federal Services, LLC  
4605 Port Chicago Hwy  
Concord, CA 94520

### CHAIN OF CUSTODY

Ref. Document # CT013 - AIR 159  
Page 1 of 4

Project Manager: Nets Johnson

Send Report To: Eddie Kalombo  
Phone/Fax Number: 415.987.0760  
Address: 4605 Port Chicago Hwy  
City: Concord, CA 94520  
eddie.kalombo@aptim.com

Project Number: 500506  
Project Name: HPNS - Parcel E-2  
Project Location: San Francisco  
Purchase Order #: 202955  
Delivery Date: 8/6/2018  
Waybill Number: N/A  
Lab Destination: CalScienc  
7440 Lincoln Way  
Garden Grove CA 92841  
Lab Contact: Terri Chang

Analyses Requested									
PEL-EPA 8082 / TO-146	PEL-EPA 8270-SM / TO-13	Asbestos (NIOSH 7400)	PM10 (40 CFR Subpt J, BAAQMD Reg 6)	TEH, M, Pb, As (40 CFR 50 App B; NIOSH 7200/60106)	Flow Rate (L/min.)	Sample Volume (m <sup>3</sup> )			
				X	1132.8	1624.44			
				X	1132.8	1624.44			
			X		1132.8	1624.44			
		X			1132.80	1624.44			
		X			2.60	2.868			
		X			2.60	2.868			
				X	1132.80	1632.36			
				X	1132.80	1627.83			

Sample ID Number		Filter No.	Collection Information			Mark	Seal	Container Type
Date	Time	Method	Mark	Seal	Container Type			
PE2-TSP073018-13UPWIND	508	07/30/18	7:15	G	A	I	8X10 EPM Whatman	
PE2-TSP073018-10ADOWNWIND	509	07/30/18	7:31	G	A	I	8X10 EPM Whatman	
PE2-PM10073018-13UPWIND	Q0373189	07/30/18	7:15	G	A	I	8X10 EPM Whatman	
PE2-PM10073018-10ADOWNWIND	Q0383190	07/30/18	7:31	G	A	I	8X10 EPM Whatman	
PE2-ASB073018-13UPWIND	DE383209	07/30/18	7:15	G	A	I	PCM	
PE2-ASB073018-10ADOWNWIND	DE383261	07/30/18	7:31	G	A	I	PCM	
PE2-TSP073118-13UPWIND	506	07/31/18	7:09	G	A	I	8X10 EPM Whatman	
PE2-TSP073118-10ADOWNWIND	507	07/31/18	7:25	G	A	I	8X10 EPM Whatman	

Temperature Blank: ☐ ☒ ☐

Special Instructions:

Turn Around Time <input type="checkbox"/> 24-hr <input type="checkbox"/> 3-5-day <input checked="" type="checkbox"/> 10-day	Level Of QC Required: <input type="checkbox"/> HI <input checked="" type="checkbox"/> Project Specific	Method Codes C = Composite G = Grab
Relinquished By: _____ Date: _____	Received By: _____ Date: _____	Matrix Codes DW = Drinking Water GW = Ground Water WW = Waste Water A = Air
Relinquished By: _____ Date: _____	Received By: _____ Date: _____	SI = Soil SL = Sludge CP = Chip Samples ASB = Asbestos, PCM Pipe, Coping



Hunters Point Naval Shipyard Procedure

Revision: 1

Approval Date: 4/23/2020

# LOW-VOLUME AIR SAMPLING PROCEDURES FOR ASBESTOS

## APPENDIX B: CHAIN OF CUSTODY (CONTINUED)

AIR MONITORING LOG										
PROJECT NAME: <b>HPNS Parcel E-2</b>				PROJ. NO. <b>506506</b>		Asbestos		TSP	PAH	PCB
STATION					COC# 001					
SAMPLE NO. <b>HP-ST13-PCB120716-UPWIND</b>					12/7/16 Station 13 Upwind					
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CEL-008	34.0	34.0	34.0	12/07/16 07:40	12/08/16 08:15	1475	1404.2	PCB	962.85	
SAMPLE NO. <b>HP-ST14-PCB120716-DOWNWIND</b>					12/7/16 Station 14 Downwind					
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CEL-074	34.0	34.0	34.0	12/07/16 07:50	12/08/16 08:30	1480	1408.0	PCB	962.88	
SAMPLE NO. <b>HP-ST13-PAH120716-UPWIND</b>					12/7/16 Station 13 Upwind					
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CEL-063 B	34.0	34.0	34.0	12/07/16 07:40	12/08/16 08:15	1475	1404.2	PAH	962.86	
SAMPLE NO. <b>HP-ST14-PAH120716-DOWNWIND</b>					12/7/16 Station 14 Downwind					
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CEL-082 T	34.0	34.0	34.0	12/07/16 07:50	12/08/16 08:30	1480	1408.0	PAH	962.88	
SAMPLE NO. <b>HP-ST13-ASBESTOS120716-UPWIND</b>					12/7/16 Station 13 Upwind					
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CO701706	2.0	2.0	2.0	12/07/16 07:40	12/08/16 08:15	1475	2.9	Asbestos	56.64	
SAMPLE NO. <b>HP-ST14-ASBESTOS120716-DOWNWIND</b>					12/7/16 Station 14 Downwind					
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
CO701678	2.0	2.0	2.0	12/07/16 07:50	12/08/16 08:30	1480	3.0	Asbestos	56.64	
SAMPLE NO. <b>HP-ST13-TSP120716-UPWIND</b>					12/7/16 Station 13 Upwind					
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
Q0364107	40.0	40.0	40.0	12/07/16 07:40	12/08/16 08:15	1475	1852.0	TSP	1132.80	
SAMPLE NO. <b>HP-ST14-TSP120716-DOWNWIND</b>					12/7/16 Station 14 Downwind					
LOT No.	FLOW RATE (CFM)			RUNNING TIME (HRS)		TOTAL TIME (min)	TOTAL VOL. (std m <sup>3</sup> )	Analysis	Flow Rate (L/min.)	
	START	STOP	AVERAGE	START	STOP					
Q0364108	40.0	40.0	40.0	12/07/16 07:50	12/08/16 08:30	1480	1897.6	TSP	1132.80	



# WORK INSTRUCTION

Work Instruction Title:	D2006-4550-011 – Operation and Use of Portable Instruments at Hunters Point Naval Shipyard	CMS Number:	N/A
Work Instruction Owner:	Federal Services HSE	Issuing Authority:	Radiation Safety

## D2006-4550-011 OPERATION AND USE OF PORTABLE INSTRUMENTS AT HUNTERS POINT NAVAL SHIPYARD

1	Minor additions to air sample requirements and sources		4/8/2020
0	Initial submittal.		3/27/2020
Rev	Changes	Approved	Date





# D2006-4550-011

## Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

<b>CMS Number:</b>	<b>Revision:</b>	<b>Approval Date:</b>
N/A	0	

### 1.0 PURPOSE

This site-specific work instruction (WI) provides specific direction for the use of portable radiological instruments at Hunters Point Naval Shipyard (HPNS) and demonstrates the suitability of the selected instruments to adequately detect the primary radionuclides of concern, radium 226 (<sup>226</sup>Ra), cesium-137 (<sup>137</sup>Cs), and strontium-90 (<sup>90</sup>Sr). The survey instruments and techniques documented herein will also be used for other potential radionuclides at HPNS, including, but not limited to, uranium-238 (<sup>238</sup>U).

### 2.0 APPLICATION

This WI provides standard practices and operating procedures for the instruments listed in Table 1 and equivalent instruments. This document provides the minimum required steps and quality checks that all employees and subcontractors are to follow when operating these instruments. Proper control, calibration, and daily checks of these instruments ensure that operating parameters demonstrate compliance with applicable data quality requirements and/or regulations. Also provided in this WI are instructions for the documentation of instrument performance and survey data. Use of these detectors is for performance of surveys to identify potential radionuclides of concern within the project workspace, on materials, or on equipment in excess of instrument specific investigation levels or project release criteria.

**Table 1. Portable Instruments**

DETECTORS	DETECTOR TYPE	INSTRUMENTS (or equivalent)		USE	Table 2 Source
Ludlum 43-37	584 cm <sup>2</sup> Gas-flow proportional	Ludlum 2360	Ludlum 2224	Alpha/beta surface contamination surveys	1,2
Ludlum 43-68	126 cm <sup>2</sup> Gas-flow proportional	Ludlum 2360	Ludlum 2224	Alpha/beta surface contamination surveys	1,2
Ludlum 43-93	100 cm <sup>2</sup> ZnS scintillator	Ludlum 2360	Ludlum 2224	Alpha/beta surface contamination surveys	1,2
Ludlum 44-10	2-in x 2-in NaI scintillator	Ludlum 2221	Ludlum 2350-1	Gamma investigation surveys	4
Ludlum 44-20	3-in x 3-in NaI scintillator	Ludlum 2221	Ludlum 2350-1	Gamma investigation surveys	4
Ludlum 43-10-1	ZnS adhered to plastic scintillator	Ludlum 2929	NA	Alpha/beta sample counting	1,2
Integral internal detector	ZnS adhered to plastic scintillator	Ludlum 3030	NA	Alpha/beta sample counting	1,2
Integral internal detector	Ion Chamber	Ludlum 9-3	NA	Beta/gamma exposure rate surveys; mR/hr	3,4
Integral internal detector	1-in x 1-in NaI scintillator	Ludlum 19	NA	Gamma exposure rate surveys; µR/hr	4
Integral internal detector	6-in x 1-in EJ-212 plastic scintillator	Ludlum 193-6	NA	Extended reach gamma exposure rate surveys; µR/hr	4



# D2006-4550-011

## Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

### 3.0 REQUIREMENTS

Implementation of this instruction requires its users to have knowledge of radiation safety principles and APTIM AMS-710-07-PR-04000, *Radiation Safety Program*.

3.1 The following personnel have responsibilities in this WI:

- Project Radiation Safety Officer (PRSO)/Alternate PRSO
- Radiological Control Supervisor (RCS)
- Lead Radiological Control Technician
- Radiological Control Technician

3.2 Responsibilities

3.2.1 The Project Radiation Safety Officer (PRSO) is responsible for the maintenance and management of the instrumentation. The PRSO is also responsible for making sure Radiological Control Technicians (RCTs) are following this procedure as described herein.

3.2.2 The Radiological Controls Supervisor (RCS) is responsible for the oversight of the RCTs operating these instruments. The lead RCT may be assigned responsibility by the PRSO or RCS for the field oversight at a specific work site of RCTs performing tasks detailed in this WI.

3.2.3 The Radiological Control Technicians (RCT) are responsible for compliance with this WI, recognizing instrumentation problems and notifying the RCS or PRSO of malfunctioning instruments. RCTs shall understand project investigation levels and release criteria and know how to compare instrument response to the applicable investigation levels or release criteria.

### 4.0 REFERENCES

CFR, Title 10, Part 20, Standards for Protection against Radiation, U.S. Government Printing Office, Washington, D.C., January 1, 2011, available online at 10CFR20 (current version).

AMS-710-07-PR-04000, *Radiation Safety Program*

AMS-710-07-WI-04005, *Radiation Safety Training*

AMS-710-07-WI-04009, *Radiological Work Permits*

AMS-710-07-WI-04012, *Radiological Surveys and Monitoring*

AMS-710-07-WI-40121, *Performing and Documenting Radiation and Contamination Surveys*

AMS-710-07-WI-04014, *Radiation Detection Instruments*

Grove Engineering, Inc., *Microshield*, current version or equivalent

SPL-SOP-8.10.2, *Radiation Monitoring Instrumentation*

U.S. Atomic Energy Commission, Regulatory Guide 1.86, *Termination of Operating Licenses for Nuclear Reactors*, Table 1.

U.S. Department of Defense, U.S. Department of Energy, U.S. Nuclear Regulatory Commission, and U.S. Environmental Protection Agency, 2000, *Multi Agency Radiation Survey and Site Investigation Manual (MARSSIM)*, NUREG 1575, Revision 1, August.

U.S. Nuclear Regulatory Commission, 2016, Code of Federal Regulations, 10 CFR 20, Standards for Protection Against Radiation.



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

<b>CMS Number:</b>	<b>Revision:</b>	<b>Approval Date:</b>
N/A	0	

U.S. Nuclear Regulatory Commission, NUREG-1757, Vol. 2, Rev. 1 "Consolidated Decommissioning Guidance, Characterization, Survey, and Determination of Radiological Criteria"

U.S. Nuclear Regulatory Commission, NUREG-1507, "Minimum Detectable Concentrations with Typical Radiation Survey Instruments for Various Contaminants and Field Conditions"

## 5.0 WORK INSTRUCTION

### 5.1 Prerequisites

- 5.1.1 Staff performing this procedure must be trained and qualified per AMS-710-07-WI-04005, Radiation Safety Training.
- 5.1.2 In collaboration with the PRSO, RCS or lead RCT, choose an appropriate and safe location for performance of instrument setups and daily function checks.
- 5.1.3 Review and sign applicable RWP(s) for the specific work area, task, or job.
- 5.1.4 Comply with general industrial safety standards and the project Accident Prevention Plan.

### 5.2 Limitations

- 5.2.1 The following limitations apply to this WI.
  - 5.2.1.1 Calibration shall be performed annually, after maintenance is performed or if the instrument fails the performance test or if proper operation is in question. Calibration is to be conducted by the manufacturer per the manufacturer's instruction manual.
  - 5.2.1.2 Table 2 compares the energies of the check sources in use to the project radionuclides of concern and demonstrates that instrument specific responses based on these check sources will result in satisfactory detection of radionuclide of concern activity.

**Table 2. Source Energy Comparison**

RADIONUCLIDE	ALPHA ENERGY (MeV)	BETA MAX ENERGY (MeV)	GAMMA ENERGY (MeV)
Radionuclides of Concern			
<sup>226</sup> Ra	4.78 (95% abun)	<sup>214</sup> Bi, 1.5 (40% abun) <sup>214</sup> Pb, 0.65 (40% abun)	<sup>226</sup> Ra, 0.18 (4% abun) <sup>214</sup> Bi, 0.69 (47% abun) <sup>214</sup> Pb, 0.35 (36% abun)
<sup>137</sup> Cs	N/A	<sup>137</sup> Cs, 0.51	<sup>137</sup> Cs, 0.662 (85% abun)
<sup>90</sup> Sr	N/A	<sup>90</sup> Sr, 0.546 (100% abun) <sup>90</sup> Y, 2.28 (99.99% abun)	N/A
<sup>238</sup> U	4.20 (76.8% abun) 4.15 (22.9% abun)	N/A	Multiple x-rays
Project Check Sources			
<sup>230</sup> Th <sup>1,2,5</sup>	4.68 (76% abun) (Source #1)		
<sup>99</sup> Tc <sup>1,3</sup>		<sup>99</sup> Tc, 0.29 (Source #2)	
<sup>137</sup> Cs <sup>1,4</sup>		<sup>137</sup> Cs, 0.51 (Source #3)	<sup>137</sup> Cs, 0.662 (85% abun) (Source #4)



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

<b>CMS Number:</b>	<b>Revision:</b>	<b>Approval Date:</b>
<b>N/A</b>	<b>0</b>	

- <sup>1</sup> The check sources in use are matched to instruments listed in Table 1.
- <sup>2</sup> <sup>230</sup>Th compares well with the alpha energy of <sup>226</sup>Ra and is slightly lower in energy so it is a conservative calibration/check source.
- <sup>3</sup> <sup>99</sup>Tc is lower in energy than the beta emissions of <sup>90</sup>Sr/<sup>90</sup>Y as well as the significant daughter products of <sup>226</sup>Ra so it is a conservative calibration/check source.
- <sup>4</sup> <sup>137</sup>Cs has a gamma which is similar to the <sup>214</sup>Pb daughter and the response of the NaI detectors has been specifically calculated to <sup>226</sup>Ra and its daughter energies using the method of NUREG-1507.
- <sup>5</sup> <sup>230</sup>Th is higher in energy than the <sup>238</sup>U alpha emissions so it is a less conservative calibration/check source. Typically, <sup>238</sup>U is determined by isotope-specific laboratory analysis.

#### 5.3 Definitions

TERM/ACRONYM	DEFINITION
Alpha Radiation ( $\alpha$ )	Alpha particles ( $\text{He}^{+2}$ ) emitted by some radionuclides while undergoing radioactive decay. While Alpha radiation does not pose an external exposure threat, alpha emitters may also emit photons (gamma or X-ray) during decay or attenuation. Alpha particles can pose an internal hazard if ingested or inhaled.
Background Radiation	Radiation that occurs naturally in the environment. Background radiation consists of cosmic radiation from outer space, or radioactive elements in geological media, building material, or other natural sources, including radon and its decay products in air and global fallout as it exists in the environment from the testing of nuclear explosive devices or from past nuclear accidents such as Chernobyl that contribute to background radiation and are not under the control of the licensee. "Background" radiation does not include radiation from source, byproduct, or special nuclear material regulated by the Nuclear Regulatory Commission (NRC).
Beta Radiation	Beta particles ( $\beta$ ) emitted by some radionuclides while undergoing radioactive decay. With few exceptions, beta-emitting radionuclides also emit photons (gamma or X-ray) during decay. Beta particles cannot penetrate human skin but do pose a hazard to the skin and lenses of the eye.
Gamma Radiation	Photons emitted from the nucleus of most atoms during the radioactive decay process. Gamma rays do not have mass or electrical charge and can be very penetrating depending on their energy level. Gamma rays pose an external hazard.
Ionizing Radiation	Alpha particles, beta particles, gamma rays, neutrons, energetic electrons or protons, and other particles capable of producing ions when interacting with matter.
Mylar	Biaxial-oriented polyethylene terephthalate (boPET) is a polyester film made from stretched polyethylene terephthalate (PET) and is used for its high tensile strength, chemical dimensional stability, transparency, reflectivity, gas and aroma barrier properties and electrical insulation.
Photomultiplier Tube (PMT)	A sensitive detector of light in the ultraviolet, visible, and near-infrared ranges of the electromagnetic spectrum. These detectors multiply the current produced by incident light by as much as 100 million times in multiple dynode stages, enabling individual photons to be detected when the incident flux of light is very low.



# D2006-4550-011

## Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

<b>CMS Number:</b>	<b>Revision:</b>	<b>Approval Date:</b>
<b>N/A</b>	<b>0</b>	

TERM/ACRONYM	DEFINITION
Scintillator	Detector or scintillation counter is used when a scintillator counter is coupled to an electronic light sensor such as a PMT or a photodiode. PMTs absorb the light emitted by the scintillator and reemit it in the form of electrons via the photoelectric effect. The subsequent multiplication of those electrons (sometimes called photo-electrons) results in an electrical pulse which can then be analyzed and yield information about the ionizing radiation that originally struck the scintillator.
Sodium Iodide (NaI)	An inorganic alkali halide salt grown into a crystal with specific geometry used to detect gamma radiation via scintillation, when coupled with a photomultiplier tube.
Zinc Sulfide (ZnS)	ZnS is utilized as a scintillation detector, because it emits light on excitation by x-rays or electron beam, making it useful for x-ray screens and cathode ray tubes. It also exhibits phosphorescence due to impurities on illumination with blue or ultraviolet light. Silver activated zinc sulfide (ZnS(Ag)) has a very high scintillation efficiency but is only available as a polycrystalline powder and is thus used in thin screens for alpha particle detection.

### 5.4 Exposure Rate Meters

Exposure rate meters (Model 19, Model 9-3) will be pre-operationally inspected and response-checked in accordance with the following steps. Both background and source response shall be verified to be within the  $\pm 20\%$  daily variability, at a minimum, at the beginning of each workday the instrument is used.

#### 5.4.1 Calibration Verification

All portable radiological instruments shall have an approved, current calibration label. Calibration verification shall be performed prior to the use of the instrument.

#### 5.4.2 Physical Check

A physical check of radiological instruments is an inspection of the general physical condition of each instrument and detector. A physical check shall be performed prior to using a radiological instrument.

The physical check should include inspecting the instrument for loose or damaged knobs, buttons, cables, and connectors, broken/damaged meter movements/displays, dented or corroded instrument cases, punctured/deformed probe/probe window(s), cables, etc., and any other physical impairments that may affect the proper operation of the instrument or detector. Any instrument or detector having a questionable physical condition shall not be used until the condition is properly corrected.

The instrument, cable, and detector as calibrated should be kept together as a unit. Do not swap components.

#### 5.4.3 Battery Check

A battery check is performed to help ensure that there is sufficient voltage being supplied to the detector and instrument circuitry for proper operation. This check shall be performed in accordance with the instrument's technical manual.



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

#### 5.4.4 High Voltage (HV) Check

The HV is adjusted during instrument calibration; additional adjustment for normal operation is not required. However, an HV check is required prior to each use in accordance with the specific instrument technical manual. For some instruments a HV check in the field is not possible. An instrument with suspected HV problems shall be immediately reported to the PRSO.

#### 5.4.5 Response Source Check

A response source check is performed to ensure that the instrument will accurately respond to a known source of radiation. Using the <sup>137</sup>Cs source, perform the response source check as follows:

Step	Action
1	Determine the background radiation level. It must be low enough to allow a measurable response to the check source being used. Careful monitoring of changing background levels is necessary to obtain accurate instrument readings.
2	Begin with the instrument on the highest range/scale and energize the audible device, if applicable.
3	Slowly move the detector towards the check source and check the instrument for an increase in audible and/or visual response.
4	If appropriate check source(s) are available, change the range/scale of the instrument to obtain a visual indication and to check each of the meter ranges/scales. If an appreciable response cannot be obtained (even in the lowest range), evaluate instrument performance by comparison to previous source check data for the instrument. Notify the Project RSO of any instrument/detector response problems. Document the response on the Ratemeter Daily Instrument Check Sheet. Plot the response on the Control Graph at the bottom of the Ratemeter Daily Instrument Check Sheet.
5	The Project RSO or designee shall set up the control graph for background and source response on the Ratemeter Daily Instrument Check Sheet such that lines indicate when an instrument is outside of the +/- 20% variability.
6	Instruments with day to day background or source responses that vary by more than 20% under identical conditions shall be removed from service and the Project RSO shall be notified.

Ratemeter instrument inspections, performance verifications, and corrective actions shall be recorded on the Ratemeter Daily Instrument Check Sheet prior to use.

### 5.5 Sample Counters

Sample counters (Model 2929, Model 3030) will be prepared for use in accordance with the following steps:

#### 5.5.1 Sample Counter Pre-Operational Requirements

Prior to the use of sample counters, the following inspections/operational verifications shall be performed in addition to those required in Section 5.4 for ratemeter type instruments (i.e., calibration verification, physical check, battery check, HV check).

#### 5.5.2 Background Measurement (Initial Project Set-Up)

The following steps shall be performed during this activity:

Step	Action
1	Ensure that the sample holder tray is empty and clean. The detector/sample holder geometry should be set up in the same configuration as that to be used when counting samples to produce the most accurate results.



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
2	Select the desired counting time. The selected time must be consistently used to perform all source and sample/swipe counting operations. The counting time directly influences the Minimum Detectable Concentration (MDC) obtained for the instrument. Although the counting time must be long enough to obtain the desired MDC, it must be short enough to be practical. The background measurements should be performed in conjunction with the MDC calculations.
3	Perform the background measurement for the selected time period (t) and record the total counts measured on the Scaler Instrumentation Check Sheet.
4	Repeat the background measurement ten times. Record the total counts observed for each measurement on the Scaler Instrumentation Check Sheet.
5	<p>Calculate the average background counts (<math>\bar{C}_b</math>) and the standard deviation (<math>SD_b</math>):</p> $\bar{C}_b = \frac{\sum_{i=1}^N C_{b_i}}{N}$ $SD_b = \sqrt{\frac{\sum_{i=1}^N (C_{b_i} - \bar{C}_b)^2}{N-1}}$ <p>Where:</p> $\sum_{i=1}^N = \text{Summation of item 1,2,3...N}$ $\bar{C}_b = \text{Average number of background counts}$ $SD_b = \text{Standard deviation of the background counts}$ $N = \text{Number of measurements}$ $C_{b_i} = \text{Background counts 1, 2, 3 ... N}$
6	Record the average background ( $\bar{C}_b$ ), background count time ( $t_b$ ), and the standard deviation ( $SD_b$ ) on the Scaler Instrumentation Check Sheet.
7	Divide $\bar{C}_b$ by $t_b$ to determine the average background count rate in cpm ( $\dot{\bar{C}}_b$ ) and record the result on the Scaler Instrumentation Check Sheet.

Background is checked, at a minimum, at the beginning of each workday the instrument is used. Acceptable background response must be within the range given by the following equation:

$$\bar{C}_b \pm 3SD_b$$

Where:

$$\bar{C}_b = \text{Average background counts.}$$

$$SD_b = \text{Standard deviation of the average background counts.}$$

If the background measurement is satisfactory, continue. If the background measurement does not meet this criterion, immediately notify the PRSO. Record the background measurement on the Scaler Daily Instrument Check Sheet.

#### 5.5.3 Source Response

Determine the detector source response with a source of known activity of a nuclide with energy decay products similar to those of the nuclide to be monitored (Table 2), as follows:

Step	Action
1	Correct the 4 pi source activity for radioactive decay (when necessary) as follows:



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
	$A = A_o e^{-\lambda T} \quad \text{Where: } \lambda = \frac{0.693}{t_{1/2}}$ <p>Where:</p> <p><math>A</math> = Present source activity.</p> <p><math>A_o</math> = Source activity at initial assay.</p> <p><math>\lambda</math> = Decay constant for the source isotope.</p> <p><math>T</math> = Time elapsed since initial source assay.</p> <p><math>t_{1/2}</math> = Source isotope half-life.</p> <p>NOTE: Time units must be consistent (days, hrs, mins., etc.)</p>
2	<p>Count the source for the same time period (<math>t_s</math>) selected during the background measurements (See Section 5.5.2, step 2).</p> <p>Initially: At project set-up or as otherwise directed by the project specific work plans or instructions, or the Project RSO, count the source ten times and calculate the average net counts (<math>\overline{C}_n</math>), the standard deviation of the average gross counts (<math>SD_g</math>), and the standard deviation of the average net source counts (<math>SD_n</math>):</p> $\overline{C}_g = \frac{\sum_{i=1}^N C_{g_i}}{N} \qquad \overline{C}_n = \overline{C}_g - \overline{C}_b$ $SD_g = \sqrt{\frac{\sum_{i=1}^N (C_{g_i} - \overline{C}_g)^2}{N - 1}} \qquad SD_n = \sqrt{(SD_g)^2 + (SD_b)^2}$ <p>Where:</p> <p><math>C_{g_i}</math> = Gross source counts (total counts observed including background) 1 through N</p> <p><math>\overline{C}_b</math> = Average background counts.</p> <p><math>\overline{C}_g</math> = Average gross counts.</p> <p><math>\overline{C}_n</math> = Average net counts.</p> <p><math>SD_n</math> = Standard deviation of the average net counts.</p> <p><math>SD_g</math> = Standard deviation of the average gross counts.</p> <p><math>N</math> = Number of measurements.</p> <p><math>SD_b</math> = Standard deviation of the average background counts.</p> <p><math>\sum_{i=1}^N</math> = Summation of item 1,2,3...N.</p> <p>Record the gross counts (<math>C_{g_i}</math>, where <math>i=1</math> to N), <math>\overline{C}_n</math> and the standard deviations (<math>SD_n</math> and <math>SD_g</math>) on the Scaler Instrumentation Check Sheet.</p>





## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action															
3	<p>Divide <math>\overline{C_n}</math> by <math>t_s</math> to determine the average net count rate (<math>\dot{C_n}</math>) and record the rate on the Scaler Instrumentation Check Sheet.</p> <p>Source response is checked daily prior to instrument use. Acceptable source response is assessed using the following equation:</p> $C_g - C_b = C_n = \overline{C_n} \pm 3SD_n$ <p>Where:</p> <table><tr><td><math>\overline{C_n}</math></td><td>=</td><td>Average net counts</td></tr><tr><td><math>C_g</math></td><td>=</td><td>Gross source counts</td></tr><tr><td><math>C_n</math></td><td>=</td><td>Net source counts</td></tr><tr><td><math>C_b</math></td><td>=</td><td>Daily background counts</td></tr><tr><td><math>SD_n</math></td><td>=</td><td>Standard deviation of the average net counts</td></tr></table> $SD_n = \sqrt{(SD_g)^2 + (SD_b)^2}$	$\overline{C_n}$	=	Average net counts	$C_g$	=	Gross source counts	$C_n$	=	Net source counts	$C_b$	=	Daily background counts	$SD_n$	=	Standard deviation of the average net counts
$\overline{C_n}$	=	Average net counts														
$C_g$	=	Gross source counts														
$C_n$	=	Net source counts														
$C_b$	=	Daily background counts														
$SD_n$	=	Standard deviation of the average net counts														
4	The Project RSO or designee shall set up the control graph for background and source response on the Sample Counter Daily Instrument Check Sheet such that lines indicate when an instrument is outside of the +/- 20% variability.															
5	Instruments with day to day background or source responses that vary by more than 20% under identical conditions shall be removed from service and the Project RSO shall be notified.															

#### 5.5.4 Instrument Efficiency ( $\epsilon$ )

Record the 4 pi detector efficiency ( $\epsilon$ ) from the instrument Calibration Certificate on the Scaler Instrumentation Check Sheet

#### 5.5.5 Calculation of Minimum Detectable Concentration (MDC)

The calculated MDC is determined to ensure that the detector being used will detect the presence of activity at or above the allowable limit under a given set of counting conditions. The MDC is the concentration that a specific instrument and technique can be expected to detect 95 percent of the time under actual conditions of use. MDC is based on the estimated detector efficiency, background, and the counting time.

MDC of each instrument shall be determined upon initial set up of the counting system and as needed following modification, calibration, repair, or replacement (i.e., new detector, cables, calibration, etc.). An MDC may be required to be adjusted based on specific materials that exhibit a different background than at initial set up.

For measurements of surface concentrations by a smear sample, the MDC<sub>static</sub> should be determined using the equation from NUREG 1507. The sample collection times should be the same as the selected background times in Section 5.5.2, step 2, if practical. The PRSO shall consult with the License RSO for all other conditions.

Step	Action
1	Calculate the beta MDC for smears in dpm/100cm <sup>2</sup> :



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
	$MDC = \frac{3 + 4.65\sqrt{\overline{C_b}}}{(\varepsilon)(t)}$ <p>Where:</p> <p><math>\overline{C_b}</math> = Average background counts during time interval <math>t</math></p> <p><math>t</math> = Sample counting time, time interval in minutes</p> <p><math>\varepsilon</math> = Efficiency for the emitted radiation</p>
2	<p>Calculate the alpha MDC for smears in dpm/100cm<sup>2</sup>:</p> $MDC = \frac{3 + 4.65\sqrt{\overline{C_b}}}{(\varepsilon)(0.8)(t)}$ <p>Where:</p> <p><math>\overline{C_b}</math> = Average background counts during time interval <math>t</math></p> <p><math>t</math> = Sample counting time, time interval in minutes</p> <p><math>\varepsilon</math> = Efficiency for the emitted radiation</p> <p>0.8 = Alpha absorption factor for air and smear sample media</p>
3	<p>Calculate the alpha air sample MDC in <math>\mu\text{Ci/mL}</math>:</p> $MDC = \frac{3 + 4.65\sqrt{\overline{C_b}}}{(\varepsilon)(Vol)(0.8)(t)(2.22E6)}$ <p>Where:</p> <p><math>\overline{C_b}</math> = Average background counts during time interval <math>t</math></p> <p><math>t</math> = Sample counting time, time interval in minutes</p> <p><math>\varepsilon</math> = Efficiency for the emitted radiation</p> <p>Vol = The volume of air collected in mL</p> <p>0.8 = Alpha absorption factor for air sample media</p> <p>2.22E6 = Converts dpm to <math>\mu\text{Ci}</math></p>
4	Record the calculated MDC values on the Scaler Instrumentation Check Sheet.

If the desired MDC cannot be attained, then inspect the instrument for equipment problems (contaminated detector or sample holder, loose cables/connectors, etc.) and notify the PRSO. If no equipment problems are found, parameters such as sample quantity, count time, or background radiation levels may have to be adjusted appropriately to obtain an acceptable MDC. An MDC that reliably satisfies the project release criteria, airborne activity requirements or remediation goals may be used. If reasonable adjustment of these parameters (as directed by the PRSO) does not result in an acceptable MDC, a more suitable instrument/detector shall be required.

Smear sample results are reported in disintegrations per minute per 100 centimeters squared (dpm/100 cm<sup>2</sup>); 2.22 dpm = 1 picocurie.

Air sample results are reported in microCuries per milliliter ( $\mu\text{Ci/mL}$ ).



# D2006-4550-011

## Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

<b>CMS Number:</b>	<b>Revision:</b>	<b>Approval Date:</b>
<b>N/A</b>	<b>0</b>	

Spreadsheet software is used to calculate instrument parameters (average background, source response, and MDC) and to track daily instrument performance checks. The PRSO or RCS will provide the instrument parameters to the RCTs via a printout or a label affixed to the meter.

### 5.5.6 Air sample counting requirements

Following sample collection, air filters will be allowed to decay for 72 hours before counting to prevent short-lived radon daughters from interfering with analysis.

Following decay, the sample filters will be counted on site using a Ludlum Model 3030 (or equivalent).

A minimum of two empty sample planchets (bias sample blanks), one sample blank count at the start of sample counting and one sample blank count upon completion of sample batch counting, will be counted with each batch of ten or less samples, PRSO/RCT shall review sample blank data sets to ensure sample blank data is background count equivalent.

Each batch of 10 or less samples shall have at least one field sample counted as a field replicate sample.

The PRSO/RCS shall review all gross alpha/beta surface wipe and air sample counting data.

Air filters indicating positive activity above action levels on initial assessment will be decayed an additional minimum of 72 hours before final on-site assay.

If activity above the action level is still indicated after the decay period, the filter will be sent to the off-site laboratory for additional analysis including low background gross alpha and beta analysis and/or isotopic analysis. The filter will be analyzed for the follow:

- Cesium-137 by gamma spectroscopy according to EPA Method 901.1MOD/DOE EML HASL 300 Method GA-01-R
- Strontium by EPA Method 905.0 or equivalent methods
- Isotopic plutonium by alpha spectroscopy
- Isotopic uranium by alpha spectroscopy
- Isotopic thorium by alpha spectroscopy
- Alpha-emitting radium isotopes by EPA Method 903.0 or equivalent methods

Upon receipt of elevate offsite air sample results, notifications will be made to the Navy.

Once all data has been received for each air sample, the PRSO/RCS will update the air sample tracking spreadsheet associated with the project routine reports to the Navy.

## 5.6 Surface Contamination Survey Instruments

Ludlum Model 2360 or Model 2224 meters with either Ludlum 43-68, Ludlum 43-93 or Ludlum 43-37 detectors will be prepared for use in accordance with the following steps:



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

#### 5.6.1 Surface Contamination Survey Instrument Preoperational Requirements

Prior to the use of these instruments and detectors, the following inspections/operational verifications shall be performed in addition to those required in Section 5.4 for ratemeter type instruments (i.e., calibration verification, physical check, battery check, HV check). Where a calculator Standard Deviation Function is used or when a spreadsheet program is used in the preoperational checks, it should be noted on the affected paperwork.

#### 5.6.2 Background Measurement (Initial Project Set up)

The following steps will be performed when conducting the initial background measurements as part of the instrument set up:

Step	Action
1	Select the desired counting time. The selected time must be consistently used to perform all source and sample counting operations. The counting time directly influences the Minimum Detectable Concentration (MDC) obtained for the instrument. Although the counting time must be long enough to obtain the desired MDC, it must be short enough to be practical. The background measurements should be performed in conjunction with the MDC calculations.
2	Perform the background measurement for the selected time period ( $t_b$ ) and record the total counts measured on the Scaler Instrumentation Check Sheet.
3	Repeat the background measurement ten times. Record the total counts observed for each measurement on the Scaler Instrumentation Check Sheet.
4	<p>Calculate the average background counts (<math>\overline{C}_b</math>) and the standard deviation (<math>SD_b</math>):</p> <div><math display="block">\overline{C}_b = \frac{\sum_{i=1}^N C_{b_i}}{N}</math><math display="block">SD_b = \sqrt{\frac{\sum_{i=1}^N (C_{b_i} - \overline{C}_b)^2}{N - 1}}</math></div> <p>Where:</p> <div><math display="block">\sum_{i=1}^N</math> = Summation of item 1,2,3...N</div> <div><math display="block">\overline{C}_b</math> = Average number of background counts</div> <div><math display="block">SD_b</math> = Standard deviation of the background counts</div> <div><math display="block">N</math> = Number of measurements</div> <div><math display="block">C_{b_i}</math> = Background counts 1, 2, 3 ... N</div>
5	Record the average background ( $\overline{C}_b$ ), background count time ( $t_b$ ), and the standard deviation ( $SD_b$ ) on the Scaler Instrumentation Check Sheet.
6	Divide $\overline{C}_b$ by $t_b$ to determine the average background count rate in cpm ( $\dot{\overline{C}}_b$ ), and record the result on the Scaler Instrumentation Check Sheet.



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Background is checked, at a minimum, at the beginning of each workday the instrument is used. Acceptable background response must be within the range determined by the following equation:

$$\overline{C}_b \pm 3SD_b$$

Where:

$C_b$  = Background counts

$\overline{C}_b$  = Average background counts.

$SD_b$  = Standard deviation of the average background counts.

If the background measurement is satisfactory, continue. If the background measurement does not meet this criterion, immediately notify the PRSO. Record the background measurement on the Scaler Daily Instrument Check Sheet.

#### 5.6.3 Source Response

Determine the detector source response with a source of known activity of a nuclide with energy decay products similar to those of the nuclide to be monitored (Table 2), as follows:

Step	Action
1	<p>Correct the source activity for radioactive decay (when necessary) as follows:</p> $A = A_o e^{-\lambda T} \quad \text{Where: } \lambda = \frac{0.693}{t_{1/2}}$ <p>Where:</p> <p><math>A</math> = Present source activity.</p> <p><math>A_o</math> = Source activity at initial assay.</p> <p><math>\lambda</math> = Decay constant for the source isotope.</p> <p><math>T</math> = Time elapsed since initial source assay.</p> <p><math>t_{1/2}</math> = Source isotope half-life.</p> <p>NOTE: Time units must be consistent (days, hrs, mins., etc.)</p>
2	<p>Count the source for the same time period (<math>t_s</math>) selected during the background measurements (See Section 5.6.2, step 2).</p> <p>Initially: At project set-up or as otherwise directed by the project specific work plans or instructions, or the Project RSO, count the source ten times and calculate the average net counts (<math>\overline{C}_n</math>), the standard deviation of the average gross counts (<math>SD_g</math>), and the standard deviation of the average net source counts (<math>SD_n</math>):</p> $\overline{C}_g = \frac{\sum_{i=1}^N C_{gi}}{N}$ $\overline{C}_n = \overline{C}_g - \overline{C}_b$ $SD_g = \sqrt{\frac{\sum_{i=1}^N (C_{gi} - \overline{C}_g)^2}{N - 1}}$ $SD_n = \sqrt{(SD_g)^2 + (SD_b)^2}$ <p>Where:</p> <p><math>C_{gi}</math> = Gross source counts (total counts observed including background) 1 through N</p> <p><math>\overline{C}_b</math> = Average background counts.</p>



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
	$\overline{C_g}$ = Average gross counts. $\overline{C_n}$ = Average net counts. $SD_n$ = Standard deviation of the average net counts. $SD_g$ = Standard deviation of the average gross counts. $N$ = Number of measurements. $SD_b$ = Standard deviation of the average background counts. $\sum_{i=1}^N$ = Summation of item 1,2,3...N.  Record the gross counts ( $C_{gi}$ , where $i=1$ to $N$ ), $\overline{C_n}$ and the standard deviations ( $SD_n$ and $SD_g$ ) on the Scaler Instrumentation Check Sheet.
3	<p>Divide <math>\overline{C_n}</math> by <math>t_s</math> to determine the average net count rate (<math>\dot{\overline{C_n}}</math>) and record the rate on the Scaler Instrumentation Check Sheet.</p> <p>Source response is checked daily prior to instrument use. Acceptable source response is assessed using the following equation:</p> $C_g - C_b = C_n = \overline{C_n} \pm 3SD_n$ <p>Where:</p> $\overline{C_n}$ = Average net counts $C_g$ = Gross source counts $C_n$ = Net source counts $C_b$ = Daily background counts $SD_n$ = Standard deviation of the average net counts $SD_n = \sqrt{(SD_g)^2 + (SD_b)^2}$

#### 5.6.4 Instrument Efficiency ( $\epsilon$ )

Record the 2 pi detector efficiency ( $\epsilon$ ) from the instrument Calibration Certificate on the Scaler Instrumentation Check Sheet.

#### 5.6.5 Calculation of Minimum Detectable Concentration (MDC)

The calculated MDC is determined to ensure that the detector being used will detect the presence of activity at or above the allowable limit under a given set of counting conditions. The MDC is the concentration that a specific instrument and technique can be expected to detect 95 percent of the time under actual conditions of use. MDC is based on the estimated detector efficiency, background, and the counting time.

MDC of each instrument shall be determined upon initial set up of the counting system and as needed following modification, calibration, repair, or replacement (i.e., new detector, cables, calibration, etc.). An MDC may be required to be determined on specific materials that exhibit a different background than at initial set up. The Project



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

RSO shall be contacted to determine if an MDC determination is necessary for specific materials.

For scanning building surfaces, the  $MDC_{scan}$  for beta should be determined using the following equation (using a value recommended in Appendix A of U.S. Nuclear Regulatory Commission, NUREG 1757, Vol. 2, Rev. 1, "Consolidated NMSS Decommissioning Guidance," for the index sensitivity  $d'$  of 1.38, which is for 95 percent detection of a concentration equal to  $MDC_{scan}$  with a 60 percent false positive). The background collection times shall be at least 1 minute, to ensure consistent data collection.

For scanning alpha, the detection probability should be determined instead of the  $MDC_{scan}$ . Further information discussed in section 5.6.6 below.

Step	Action
1	<p>Calculate the beta <math>MDC_{scan}</math> in dpm/100cm<sup>2</sup>:</p> $MDC_{scan}(surfaces) = \frac{1.38 \sqrt{C_{bscan}}}{(\sqrt{p})(\epsilon)(t_{scan})(A/100 cm^2)}$ <p>Where:</p> <p>1.38 = Index of sensitivity <math>d'</math></p> <p><math>C_{bscan}</math> = Average background counts in time interval <math>t_{scan}</math></p> <p><math>p</math> = Surveyor efficiency (0.5)</p> <p><math>\epsilon</math> = Instrument 2 pi efficiency (x surface efficiency of 0.5) for the emitted radiation</p> <p><math>t_{scan}</math> = Sample count time, time interval of the observation while the probe passes over the source <b>in minutes</b>.</p> <p><math>A</math> = Active area of probe in cm<sup>2</sup></p>
2	Record the calculated beta $MDC_{scan}$ on the Scaler Instrumentation Check Sheet.
3	For static measurements of surface concentrations by either direct measurement or by a smear sample, the $MDC_{static}$ should be determined using the equation from NUREG 1507. The sample collection times should be the same as the selected background times in Section 5.6.2, step 2, if practical. The Project RSO shall consult with the License RSO for all other conditions.
4	<p>Calculate the <math>MDC_{static}</math> in dpm/100cm<sup>2</sup>:</p> $MDC_{static} = \frac{3 + 4.65 \sqrt{C_{bstatic}}}{(K)(t_{static})} = \frac{3 + 4.65 \sqrt{C_{bstatic}}}{(\epsilon)(A/100 cm^2)(t_{static})}$ <p>Where:</p> <p><math>C_{bstatic}</math> = Average background counts during time interval <math>t_{static}</math></p> <p><math>t_{static}</math> = Sample counting time, time interval in min. the probe is in direct contact with the surface or smear</p> <p><math>K</math> = <math>\epsilon^*(A/100 cm^2)</math> A calibration constant (best estimate) to convert counts/min to dpm/100cm<sup>2</sup>.</p> <p><math>A</math> = Probe's active area, in cm<sup>2</sup></p>



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
	$\epsilon$ = Instrument 2 pi efficiency (x surface efficiency of 0.5) for the emitted radiation
5	Record the calculated $MDC_{static}$ on the Scaler Instrumentation Check Sheet.

The calculated  $MDC_{static}$  should be less than 50 percent of the appropriate project release criterion or remediation goal, and while there is no specific recommendation of  $MDC_{scan}$ , it should be no more than 50 percent of the appropriate project release criterion or remediation goal if possible. If the desired MDC cannot be attained, then inspect the instrument for equipment problems (contaminated detector or sample holder, loose cables/connectors, etc.) and notify the Project RSO. If no equipment problems are found, parameters such as sample quantity, count time, or background radiation levels may have to be adjusted appropriately to obtain an acceptable MDC. On a case by case basis an MDC that is greater than 50 percent but less than 90 percent of the project release criteria or remediation goals may be used with the concurrence of the License RSO. If reasonable adjustment of these parameters (as directed by the Project RSO) does not result in an acceptable MDC, a more suitable instrument/detector shall be used.

Static counts are reported in dpm/100 cm<sup>2</sup>; 2.22 dpm = 1 picocurie.

Spreadsheet software is used to calculate instrument parameters (average background, efficiency, and MDC) and to track daily instrument performance checks. The PRSO or RCS will provide the instrument parameters to the RCTs via a printout or a label affixed to the meter.

#### 5.6.6 Count Detection Probability for Alpha Scans

In accordance with the adopted process from the RASO guidance document *Conducting Alpha Scans for Radium*, the background should be evaluated to determine the applicable equation to be used to calculate the alpha count detection probability. The expected background response of alpha detectors varies from near zero to 10 cpm. Following sections cover to determine the alpha count detection probability of each instrument.

Step	Action
1	<p><b>Background counts <math>\leq 5</math> cpm;</b></p> <p>For alpha survey instrumentation with background counts less than or equal to 5 cpm, a single count provides a surveyor sufficient cause to stop and investigate further. Assuming this to be true, the probability of detecting given levels of alpha surface contamination can be calculated by use of Poisson summation statistics.</p> <p>Given a known scan rate and a surface contamination release limit, the probability of detecting a single count while passing over the contaminated area is given by the following equation:</p> $P(n \geq 1) = 1 - e^{-\frac{(G)(\epsilon)(\epsilon_p)(d)}{(60)(n)}}$





## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
	<p>Where:</p> $P(n \geq 1) = \text{probability of observing a single count}$ $G = \text{surface contamination release limit (dpm)}$ $\varepsilon_i = \text{detector efficiency (2}\pi\text{)}$ $\varepsilon_s = \text{surface efficiency 0.25}$ $d = \text{width of detector in direction of scan (cm)}$ $v = \text{scan speed (centimeters per second)}$ <p>A probability of detection should be as close as practicable to 90%, but shall not be lower than 68%.</p> <p><b>Background counts &gt; 5 cpm;</b></p> <p>The larger (584 cm<sup>2</sup>) gas proportional detectors have alpha background count rates on the order of 1 cpm to 10 cpm. If the background is less than or equal to 5 cpm, a single count will not cause a surveyor to investigate further. A counting period long enough to establish that a single count indicates an elevated contamination level would be prohibitively inefficient. For an instrument with lower background, the surveyor usually will need to get at least two counts while passing over the source area before stopping for further investigation.</p> <p>Assuming this assumption is valid; the probability of getting two or more counts can be calculated by equation:</p> $P(n \geq 2) = 1 - \left[ 1 + \frac{(G\varepsilon_i\varepsilon_s + B)t}{60} \right] \left[ e^{\frac{-(G\varepsilon_i\varepsilon_s + B)t}{60}} \right]$ <p>Where:</p> $P(n \geq 2) = \text{probability of getting two or more counts during the time interval } t$ $t = \text{time interval of detector over source (seconds)}$ $G = \text{surface contamination release limit (dpm)}$ $\varepsilon_i = \text{detector efficiency (2}\pi\text{)}$ $\varepsilon_s = \text{surface efficiency 0.25}$ $B = \text{background count rate (cpm)}$ <p>A probability of detection should be as close as practicable to 90% but shall not be lower than 68%.</p> <p>Once a count is recorded and the guideline level of contamination is present, the</p>



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
	<p>surveyor should stop and wait until the probability of getting another count is at least 68 percent. This time interval can be calculated by the following equation:</p> $t = \frac{13,800}{CAE}$ <p>Where:</p> <p style="margin-left: 40px;"><math>t</math> = time period for static count(s)</p> <p style="margin-left: 40px;"><math>C</math> = surface contamination release limit (dpm/100 cm<sup>2</sup>)</p> <p style="margin-left: 40px;"><math>A</math> = physical probe area (cm<sup>2</sup>)</p> <p style="margin-left: 40px;"><math>E</math> = detector 2 pi efficiency (x surface efficiency)</p> <p>Alpha scan for the Ludlum 43-93 (or equivalent) detector and/or the Ludlum 43-37 (or equivalent) detector must be sufficient for the conditions to be met.</p> <p>The PRSO or RCS will determine the time period required for each instrument specific probability time interval, and document it on a printout or a label applied to the meter.</p>
2	Record the calculated alpha count detection probability and the time period on the Scaler Instrumentation Check Sheet.

#### 5.7 Gamma Survey Instruments

Ludlum Model 2221 or equivalent meters with either Ludlum 44-10 or Ludlum 44-20 detectors are only used to identify potential anomalous gamma count rates due to the presence of low-level radiological objects or soil contamination (resulting from either direct gamma emissions or bremsstrahlung due to beta emissions). Count rate measurements acquired using the 44-10 and 44-20 are not used in determining soil activity concentrations of radionuclides of concern but may be used to develop estimates for potential exposure or health risk.

Ludlum Model 2221 or equivalent meters with either Ludlum 44-10 or Ludlum 44-20 detectors will be prepared for use in accordance with the following steps.

##### 5.7.1 Background Determination

The following steps will be used in determining background count rates for gamma scintillation detectors during instrument quality control checks:

Step	Action
1	Set the counting time to one minute.
2	Perform the background measurement for the selected time period ( $t_b$ ) and record the total counts measured.



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
3	For initial instrument setup, repeat the background measurement ten times. Record the total counts observed for each measurement.
4	<p>Calculate the average background counts (<math>\overline{C_b}</math>)</p> $\overline{C_b} = \frac{\sum_{i=1}^N C_{b_i}}{N}$ <p>Where:</p> <p><math>\sum_{i=1}^N</math> = Summation of item 1,2,3...N</p> <p><math>\overline{C_b}</math> = Average number of background counts</p> <p><math>N</math> = Number of measurements</p> <p><math>C_{b_i}</math> = Background counts 1, 2, 3 ... N</p>
5	Divide $\overline{C_b}$ by $t_b$ to determine the average background count rate in cpm ( $\dot{\overline{C_b}}$ ) and record the result.

Daily: perform a single background count ( $C_b$ ) prior to instrument use. This value must be within the range determined by using the following formula:

$$\overline{C_b} \pm 20\%$$

Where:

$$\overline{C_b} = \text{Average background counts}$$

Record the background measurement. If the background measurement is satisfactory, continue. If the background measurement does not meet this criterion, immediately notify the PRSO.

#### 5.7.2 Source Response

The following steps will be used in determining background count rates for gamma scintillation detectors during instrument quality control checks:

Step	Action
1	Set the counting time to one minute.
2	Place the $^{137}\text{Cs}$ check source under the source check jig and the detector into the jig, ensuring the source to detector distance is 10 cm (4 in).
3	Perform the source measurement for the selected time period ( $t_b$ ) and record the total counts measured.
4	For initial instrument setup, repeat the source measurement ten times. Record the total counts observed for each measurement.
5	Calculate the average background counts ( $\overline{C_s}$ )



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

Step	Action
	$\overline{C_s} = \frac{\sum_{i=1}^N C_{s_i}}{N}$ <p>Where:</p> $\sum_{i=1}^N = \text{Summation of item 1,2,3...N}$ $\overline{C_s} = \text{Average number of source counts}$ $N = \text{Number of measurements}$ $C_{s_i} = \text{Source counts 1, 2, 3 ... N}$
6	<p>Divide <math>\overline{C_s}</math> by <math>t</math> to determine the average source count rate in cpm (<math>\dot{C_s}</math>) and record the result.</p>

Daily: prior to instrument use, perform a single source count ( $C_s$ ) in the same geometry employed for determining the initial source response. This value must fall within the range determined by using the following formula:

$$\overline{C_s} \pm 20\%$$

Where:

$$\overline{C_s} = \text{Average source counts}$$

Record the source measurement. If the source measurement is satisfactory, continue. If the source measurement does not meet this criterion, immediately notify the PRSO.

#### 5.7.3 Reference Area Background Determination

Initially, for each gamma survey instrument designated for soil investigation use, perform a survey of the designated reference area identified by the PRSO in consultation with the client in accordance with AMS-710-07-WI-40122 "Gamma Walkover Survey Using Global Positioning System" or other project-specific survey method.

Data collection will include scan data for the entire reference area and a minimum of 20 1-minute static counts collected within the reference area at a probe height of 10 cm. The location of the static counts will be determined by applying a systematic grid over the reference area using a random starting coordinate.

The scan data and the static measurements will be compiled and a mean count rate for static counts will be determined using spreadsheet and scan counts will be determined using the GIS software, and the standard deviation for each will be calculated.

Investigation levels for scans (mean of the scan data set plus 3 standard deviations) and investigation levels for static measurements (mean of the static data set plus 3 standard deviations) will be calculated for each gamma survey instrument in use. The PRSO or RCS will provide the RCTs with the instrument parameters and investigation levels on a spreadsheet printout or a label affixed to the meter.



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

#### 5.7.4 Performance of Gamma Surveys

Surveys with the Ludlum 44-10, Ludlum 44-20 detectors, or the RS-700 detectors are used to identify areas of elevated gamma counts that may be the result of radiological objects or contamination. Locations identified during scanning that exceed the instrument specific investigation level (as described in the project-specific work plans) require further investigation. Investigations may consist of reacquiring the elevated location and conducting a static count. Also, the RCT will rescan the nearby vicinity to assess if the elevated reading is the result of a point source or distributed radioactive material. If the static count is less than the static investigation level and there is no evidence of a point source, further investigation is not required. If the static investigation level is exceeded or there is evidence of distributed contamination at or above either investigation level the area will be flagged for further intrusive investigation.

#### 5.7.5 Calculation of Gamma Scan Minimum Detectable Concentration (MDC)

The following presents an example calculation of minimum detectable count rate and corresponding scan MDC for soil. Additional calculations should be performed as necessary as conditions change to confirm expected scan sensitivities.

The minimum detectable number of net source counts in the scan interval, for an ideal observer, can be determined by multiplying the square root of the number of background counts ( $b_i$ ) (in the scan interval) by the detectability value ( $d'$ ) associated with the desired performance as shown as follows:

$$MDCR = d' \sqrt{(b_i)} \left( \frac{60}{i} \right)$$

Where:

- $d'$  = index of sensitivity ( $\alpha$  and  $\beta$  error)
- $b_i$  = number of background counts in scan time interval (count)
- $i$  = scan or observation interval(s)

The following is an example calculation of the minimum detectable count rate (MDCR) in cpm for a typical Ludlum Model 2221/44-10 setup. It was noted that a typical source remained under the probe for 2 seconds during the scan; therefore, the average number of background counts of 6,378 cpm (reference area mean count rate for all scan instruments used) in the observation interval of 2 seconds was 212.6 cpm ( $b_i = 6,378 \times [2/60]$ ).

The performance goal for scanning surveys requires a rate of 95 percent for true positives and 5 percent for false positives. From Table 6.5 of MARSSIM (DoD et al., 2000), the value of  $d'$ , representing this performance goal, is 3.28.

Using these inputs, the MDCR is:

$$MDCR = 3.28 \sqrt{(212.6)} \left( \frac{60}{2} \right) = 1435 \text{ cpm}$$

The MDCR including surveyor efficiency ( $MDCR_{Surveyor}$ ) can be calculated using a surveyor efficiency ( $P$ ) of 0.50 and using the following equation:

$$MDCR_{Surveyor} = \frac{MDCR}{\sqrt{p}} = \frac{1435}{\sqrt{0.50}} = 2029 \text{ cpm}$$

The gamma scan MDC (in pCi/g) for land areas is based on the area of elevated activity, depth of contamination, and the radionuclide (i.e., energy and yield of gamma emissions). To establish the scan MDC, the relationship between the detector's net



## D2006-4550-011

### Operation and Use of Portable Instruments at Hunters Point Naval Shipyard

CMS Number:	Revision:	Approval Date:
N/A	0	

count rate to net exposure rate must be established. The initial step in this process is determining the  $MDCR_{Surveyor}$  as shown in preceding paragraphs.

The corresponding minimum detectable exposure rate (MDER) is determined for a 2-inch by 2-inch NaI probe and the ROC. Manufacturer specifications for the Ludlum 44-10 indicate a cpm to microrentgen per hour ( $\mu R/hr$ ) rate of 900 for  $^{137}Cs$ . This value converts to a calibration constant of  $5.4E10$  counts per R. An exposure rate can be calculated for a given count rate and vice versa using the following equation:

$$MDER \left( \frac{\mu R}{hr} \right) = \frac{MDCR_{Surveyor} \times 6E07}{cc}$$

Where:

$MDCR_{Surveyor}$  = 2029 cpm

cc = calibration constant =  $5.4E10$

6E07 = conversion factor accounting for differences in time and activity units ( $\mu R\text{-min}/(R\text{-hr})$ )

The MDER for  $^{137}Cs$  and  $^{226}Ra$  calculated in the manner described above is  $2.25 \mu R/hr$ .

Modeling (using MicroShield Version 5.05 [Grove Engineering, 1996] or similar software) is used to determine the net exposure rate produced by  $1.0 \text{ pCi/g}$  of  $^{226}Ra$  and its daughter products after 30 years of ingrowth in soil.

- The physical and geometrical factors considered in the modeling included:
- The exposure point of 4 inches above the soil was used.
- The density of 1.6 grams per cubic centimeter was used for soil.
- The depth of the area of elevated activity was 15 centimeters.
- The circular dimension of the cylindrical area of elevated activity was  $0.25 \text{ m}^2$ .

Using the above input parameters, MicroShield Version 5.05 (Grove Engineering, 1996) calculates the exposure rate to be  $0.7384 \mu R/hr$  for  $^{226}Ra$  (accounting for buildup). The radionuclide concentration of  $^{226}Ra$  necessary to yield the MDER of  $2.25 \mu R/hr$  is the scan MDC and may be calculated as follows:

$$^{226}Ra \text{ Scan MDC} = \frac{1.0 \frac{\text{pCi}}{\text{g}} \times 2.25 \frac{\mu R}{hr}}{0.7384 \frac{\mu R}{hr}} = 3.05 \text{ pCi/g}$$

This value is consistent with the default value of  $2.80 \text{ pCi/g}$  for a  $^{226}Ra$  soil source reported in NUREG-1507.

Site-specific scan MDCs were not modeled for  $^{137}Cs$ ; rather the default value from NUREG-1507 is used. The reported default scan MDC for a 2 by 2 NaI from NUREG-1507 is  $6.4 \text{ pCi/g}$  for  $^{137}Cs$ .

## 6.0 ATTACHMENTS

None



STATE WATER RESOURCES CONTROL BOARD  
REGIONAL WATER QUALITY CONTROL BOARDS

CALIFORNIA STATE



ENVIRONMENTAL LABORATORY ACCREDITATION PROGRAM

**CERTIFICATE OF ENVIRONMENTAL ACCREDITATION**

Is hereby granted to

**Eurofins Calscience, LLC**

7440 Lincoln Way

Garden Grove, CA 92841-1427

Scope of the certificate is limited to the  
"Fields of Testing"  
which accompany this Certificate.

Continued accredited status depends on successful completion of on-site inspection,  
proficiency testing studies, and payment of applicable fees.

This Certificate is granted in accordance with provisions of  
Section 100825, et seq. of the Health and Safety Code.

Certificate No.: **2944**

Expiration Date: **9/30/2020**

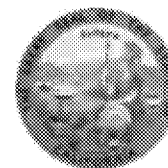
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Christine Sotelo, Chief  
Environmental Laboratory Accreditation Program



**CALIFORNIA STATE  
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Phone: 7148955494

**Certificate No. 2944**  
**Expiration Date 9/30/2020**

**Field of Testing: 102 - Inorganic Chemistry of Drinking Water**

102.015	001	Hydrogen Ion (pH)	EPA 150.1
102.026	001	Calcium	EPA 200.7
102.026	002	Magnesium	EPA 200.7
102.026	003	Potassium	EPA 200.7
102.026	004	Silica	EPA 200.7
102.026	005	Sodium	EPA 200.7
102.026	006	Hardness (Calculation)	EPA 200.7
102.030	001	Bromide	EPA 300.0
102.030	003	Chloride	EPA 300.0
102.030	005	Fluoride	EPA 300.0
102.030	006	Nitrate (as N)	EPA 300.0
102.030	007	Nitrite (as N)	EPA 300.0
102.030	008	Phosphate,Ortho (as P)	EPA 300.0
102.030	009	Sulfate (as SO4)	EPA 300.0
102.040	003	Chlorate	EPA 300.1
102.045	001	Perchlorate	EPA 314.0
102.047	001	Perchlorate	EPA 331.0
102.070	001	Phosphate,Ortho (as P)	EPA 365.1
102.100	001	Alkalinity	SM 2320 B-1997
102.121	001	Hardness	SM 2340 C-1997
102.130	001	Specific Conductance	SM 2510 B-1997
102.140	001	Residue, Filterable TDS	SM 2540 C-1997
102.148	001	Calcium	SM 3500-Ca B-1997
102.174	001	Chlorine, Free	SM 4500-Cl F-2000
102.174	002	Chlorine, Total Residual	SM 4500-Cl F-2000
102.175	001	Chlorine, Free	SM 4500-Cl G-2000
102.175	002	Chlorine, Total Residual	SM 4500-Cl G-2000
102.190	001	Cyanide, Total	SM 4500-CN E-1999
102.200	001	Fluoride	SM 4500-F C-2011
102.203	001	Hydrogen Ion (pH)	SM 4500-H+ B-2000
102.220	001	Nitrite (as N)	SM 4500-NO2 B-2000
102.240	001	Phosphate,Ortho (as P)	SM 4500-P E-1999
102.260	001	Total Organic Carbon TOC	SM 5310 B-2000

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Page 1 of 7



**Eurofins Calscience, LLC**

**Certificate No.:** 2944

**Expiration Date:** 9/30/2020

102.261	001	Dissolved Organic Carbon (DOC)	SM 5310 B-2000
102.264	001	Total Organic Carbon TOC	SM 5310 D-2000
102.265	001	Dissolved Organic Carbon (DOC)	SM 5310 D-2000
<b>Field of Testing: 103 - Toxic Chemical Elements of Drinking Water</b>			
103.130	001	Aluminum	EPA 200.7
103.130	003	Barium	EPA 200.7
103.130	004	Beryllium	EPA 200.7
103.130	005	Cadmium	EPA 200.7
103.130	007	Chromium	EPA 200.7
103.130	008	Copper	EPA 200.7
103.130	009	Iron	EPA 200.7
103.130	011	Manganese	EPA 200.7
103.130	012	Nickel	EPA 200.7
103.130	015	Silver	EPA 200.7
103.130	017	Zinc	EPA 200.7
103.130	018	Boron	EPA 200.7
103.140	001	Aluminum	EPA 200.8
103.140	002	Antimony	EPA 200.8
103.140	003	Arsenic	EPA 200.8
103.140	004	Barium	EPA 200.8
103.140	005	Beryllium	EPA 200.8
103.140	006	Cadmium	EPA 200.8
103.140	007	Chromium	EPA 200.8
103.140	008	Copper	EPA 200.8
103.140	009	Lead	EPA 200.8
103.140	010	Manganese	EPA 200.8
103.140	012	Nickel	EPA 200.8
103.140	013	Selenium	EPA 200.8
103.140	014	Silver	EPA 200.8
103.140	015	Thallium	EPA 200.8
103.140	016	Zinc	EPA 200.8
103.140	017	Boron	EPA 200.8
103.140	018	Vanadium	EPA 200.8
103.160	001	Mercury	EPA 245.1
103.310	001	Chromium (VI)	EPA 218.6
<b>Field of Testing: 104 - Volatile Organic Chemistry of Drinking Water</b>			
104.030	001	1,2-Dibromoethane (EDB, Ethylene Dibromide)	EPA 504.1
104.030	002	1,2-Dibromo-3-chloropropane (DBCP)	EPA 504.1
104.035	001	1,2,3-Trichloropropane (TCP)	SRL 524M-TCP
104.040	000	Volatile Organic Compounds	EPA 524.2
<b>Field of Testing: 108 - Inorganic Constituents in Non-Potable Water</b>			
108.020	001	Specific Conductance	EPA 120.1

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108.110	001	Turbidity	EPA 180.1
108.112	001	Boron	EPA 200.7
108.112	002	Calcium	EPA 200.7
108.112	003	Hardness (Calculation)	EPA 200.7
108.112	004	Magnesium	EPA 200.7
108.112	005	Potassium	EPA 200.7
108.112	006	Silica, Dissolved	EPA 200.7
108.112	007	Sodium	EPA 200.7
108.113	001	Boron	EPA 200.8
108.120	001	Bromide	EPA 300.0
108.120	002	Chloride	EPA 300.0
108.120	003	Fluoride	EPA 300.0
108.120	008	Sulfate (as SO <sub>4</sub> )	EPA 300.0
108.120	012	Nitrate (as N)	EPA 300.0
108.120	013	Nitrate-Nitrite (as N)	EPA 300.0
108.120	014	Nitrite (as N)	EPA 300.0
108.120	015	Phosphate, Ortho (as P)	EPA 300.0
108.209	001	Ammonia (as N)	EPA 350.1
108.211	002	Kjeldahl Nitrogen, Total (as N)	EPA 351.2
108.260	001	Phosphate, Ortho	EPA 365.1
108.261	001	Phosphorus, Total	EPA 365.1
108.264	001	Phosphate, Ortho	EPA 365.3
108.265	001	Phosphorus, Total	EPA 365.3
108.323	001	Chemical Oxygen Demand	EPA 410.4
108.360	001	Phenols, Total	EPA 420.1
108.381	001	Oil & Grease Total	EPA 1664 A
108.385	001	Color	SM 2120 B-2001
108.390	001	Turbidity	SM 2130 B-2001
108.410	001	Alkalinity	SM 2320 B-1997
108.421	001	Hardness	SM 2340 C-1997
108.430	001	Specific Conductance	SM 2510 B-1997
108.439	001	Residue, Volatile	SM 2540 E-1997
108.440	001	Residue, Total	SM 2540 B-1997
108.441	001	Residue, Filterable TDS	SM 2540 C-1997
108.442	001	Residue, Non-filterable TSS	SM 2540 D-1997
108.443	001	Residue, Settleable	SM 2540 F-1997
108.451	001	Chloride	SM 4500-Chloride C-1997
108.464	001	Chlorine, Total Residual	SM 4500-Cl F-2000
108.464	002	Chlorine, Free	SM 4500-Cl F-2000
108.472	001	Cyanide, Total	SM 4500-CN E-1999
108.473	001	Cyanide, Amenable	SM 4500-CN G-1999
108.480	001	Fluoride	SM 4500-F B,C-1997

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Page 3 of 7

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**Certificate No.:** 2944

**Expiration Date:** 9/30/2020

108.490	001	Hydrogen Ion (pH)	SM 4500-H+ B-2000
108.500	002	Ammonia (as N)	SM 4500-NH3 B,C-1997
108.501	002	Kjeldahl Nitrogen, Total (as N)	SM 4500-NH3 C-1997
108.504	002	Ammonia (as N)	SM 4500-NH3 F-1997
108.511	001	Kjeldahl Nitrogen, Total (as N)	SM 4500-Norg B- 1997
108.514	001	Nitrite (as N)	SM 4500-NO2 B-2000
108.528	001	Nitrate-Nitrite (as N)	SM 4500-NO3- E-2000
108.536	001	Oxygen, Dissolved	SM 4500-O G-2001
108.584	001	Sulfide (as S)	SM 4500-S D-2000
108.592	001	Biochemical Oxygen Demand	SM 5210 B -2001
108.592	002	Carbonaceous BOD	SM 5210 B -2001
108.595	001	Chemical Oxygen Demand	SM 5220 D-1997
108.598	001	Organic Carbon-Total (TOC)	SM 5310 D-2000
108.605	001	Surfactants	SM 5540 C-2000
108.626	001	Phenols, Total	SM 5530 D-2010

**Field of Testing:** 109 - Metals and Trace Elements in Non-Potable Water

109.010	001	Aluminum	EPA 200.7
109.010	002	Antimony	EPA 200.7
109.010	003	Arsenic	EPA 200.7
109.010	004	Barium	EPA 200.7
109.010	005	Beryllium	EPA 200.7
109.010	006	Boron	EPA 200.7
109.010	007	Cadmium	EPA 200.7
109.010	009	Chromium	EPA 200.7
109.010	010	Cobalt	EPA 200.7
109.010	011	Copper	EPA 200.7
109.010	012	Iron	EPA 200.7
109.010	013	Lead	EPA 200.7
109.010	015	Manganese	EPA 200.7
109.010	016	Molybdenum	EPA 200.7
109.010	017	Nickel	EPA 200.7
109.010	019	Selenium	EPA 200.7
109.010	021	Silver	EPA 200.7
109.010	023	Thallium	EPA 200.7
109.010	024	Tin	EPA 200.7
109.010	025	Titanium	EPA 200.7
109.010	026	Vanadium	EPA 200.7
109.010	027	Zinc	EPA 200.7
109.020	001	Aluminum	EPA 200.8
109.020	002	Antimony	EPA 200.8
109.020	003	Arsenic	EPA 200.8
109.020	004	Barium	EPA 200.8

As of 10/3/2019 , this list supersedes all previous lists for this certificate number.  
Customers: Please verify the current accreditation standing with the State.

**Eurofins Calscience, LLC**

**Certificate No.:** 2944

**Expiration Date:** 9/30/2020

109.020	005	Beryllium	EPA 200.8
109.020	006	Cadmium	EPA 200.8
109.020	007	Chromium	EPA 200.8
109.020	008	Cobalt	EPA 200.8
109.020	009	Copper	EPA 200.8
109.020	010	Lead	EPA 200.8
109.020	011	Manganese	EPA 200.8
109.020	012	Molybdenum	EPA 200.8
109.020	013	Nickel	EPA 200.8
109.020	014	Selenium	EPA 200.8
109.020	015	Silver	EPA 200.8
109.020	016	Thallium	EPA 200.8
109.020	017	Vanadium	EPA 200.8
109.020	018	Zinc	EPA 200.8
109.020	021	Iron	EPA 200.8
109.020	022	Tin	EPA 200.8
109.020	023	Titanium	EPA 200.8
109.104	001	Chromium (VI)	EPA 218.6
109.190	001	Mercury	EPA 245.1

**Field of Testing:** 110 - Volatile Organic Constituents in Non-Potable Water

110.020	000	Purgeable Aromatics	EPA 602
110.040	000	Purgeable Organic Compounds	EPA 624

**Field of Testing:** 111 - Semi-volatile Organic Constituents in Non-Potable Water

111.060	000	Polynuclear Aromatics	EPA 610
111.100	000	Base/Neutral & Acid Organics	EPA 625
111.170	000	Organochlorine Pesticides and PCBs	EPA 608

**Field of Testing:** 114 - Inorganic Chemistry of Hazardous Waste

114.010	001	Antimony	EPA 6010 B
114.010	002	Arsenic	EPA 6010 B
114.010	003	Barium	EPA 6010 B
114.010	004	Beryllium	EPA 6010 B
114.010	005	Cadmium	EPA 6010 B
114.010	006	Chromium	EPA 6010 B
114.010	007	Cobalt	EPA 6010 B
114.010	008	Copper	EPA 6010 B
114.010	009	Lead	EPA 6010 B
114.010	010	Molybdenum	EPA 6010 B
114.010	011	Nickel	EPA 6010 B
114.010	012	Selenium	EPA 6010 B
114.010	013	Silver	EPA 6010 B
114.010	014	Thallium	EPA 6010 B
114.010	015	Vanadium	EPA 6010 B

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**Expiration Date:** 9/30/2020

114.010	016	Zinc	EPA 6010 B
114.020	001	Antimony	EPA 6020
114.020	002	Arsenic	EPA 6020
114.020	003	Barium	EPA 6020
114.020	004	Beryllium	EPA 6020
114.020	005	Cadmium	EPA 6020
114.020	006	Chromium	EPA 6020
114.020	007	Cobalt	EPA 6020
114.020	008	Copper	EPA 6020
114.020	009	Lead	EPA 6020
114.020	010	Molybdenum	EPA 6020
114.020	011	Nickel	EPA 6020
114.020	012	Selenium	EPA 6020
114.020	013	Silver	EPA 6020
114.020	014	Thallium	EPA 6020
114.020	015	Vanadium	EPA 6020
114.020	016	Zinc	EPA 6020
114.103	001	Chromium (VI)	EPA 7196 A
114.106	001	Chromium (VI)	EPA 7199
114.130	001	Lead	EPA 7420
114.140	001	Mercury	EPA 7470 A
114.141	001	Mercury	EPA 7471 A
114.241	001	Corrosivity - pH Determination	EPA 9045 C
114.250	001	Fluoride	EPA 9056

**Field of Testing: 115 - Extraction Test of Hazardous Waste**

115.020	001	Toxicity Characteristic Leaching Procedure (TCLP)	EPA 1311 (TCLP)
115.021	001	TCLP Inorganics	EPA 1311 (TCLP)
115.022	001	TCLP Extractables	EPA 1311 (TCLP)
115.023	001	TCLP Volatiles	EPA 1311 (TCLP)
115.030	001	Waste Extraction Test (WET)	CCR Chapter 11, Article 5, Appendix II
115.040	001	Synthetic Precipitation Leaching Procedure (SPLP)	EPA 1312 (SPLP)

**Field of Testing: 116 - Volatile Organic Chemistry of Hazardous Waste**

116.030	001	Gasoline-range Organics	EPA 8015 B
116.040	041	Methyl tert-butyl Ether (MTBE)	EPA 8021 B
116.040	061	Aromatic Volatiles	EPA 8021 B
116.080	000	Volatile Organic Compounds	EPA 8260 B
116.080	120	Oxygenates	EPA 8260 B
116.100	001	Total Petroleum Hydrocarbons - Gasoline (GRO)	LUFT GC/MS
116.100	010	BTEX and MTBE	LUFT GC/MS
116.110	001	Total Petroleum Hydrocarbons - Gasoline (GRO)	LUFT

**Field of Testing: 117 - Semi-volatile Organic Chemistry of Hazardous Waste**

117.010	001	Diesel-range Total Petroleum Hydrocarbons	EPA 8015 B
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As of 10/3/2019, this list supersedes all previous lists for this certificate number.  
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**Eurofins Calscience, LLC**

**Certificate No.:** 2944

**Expiration Date:** 9/30/2020

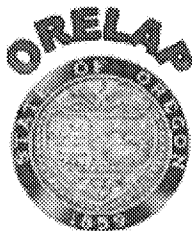
117.016	001	Diesel-range Total Petroleum Hydrocarbons	LUFT
117.110	000	Extractable Organics	EPA 8270 C
117.140	000	Polynuclear Aromatic Hydrocarbons	EPA 8310
117.170	000	Nitroaromatics and Nitramines	EPA 8330
117.210	000	Organochlorine Pesticides	EPA 8081 A
117.220	000	PCBs	EPA 8082
117.240	000	Organophosphorus Pesticides	EPA 8141 A
117.250	000	Chlorinated Herbicides	EPA 8151 A

**Field of Testing: 120 - Physical Properties of Hazardous Waste**

120.010	001	Ignitability	EPA 1010
120.040	001	Reactive Cyanide	Section 7.3 SW-846
120.050	001	Reactive Sulfide	Section 7.3 SW-846
120.080	001	Corrosivity - pH Determination	EPA 9045 C

As of 10/3/2019, this list supersedes all previous lists for this certificate number.  
Customers: Please verify the current accreditation standing with the State.

Page 7 of 7



# OREGON

## Environmental Laboratory Accreditation Program



NELAP Recognized

**Eurofins Calscience, LLC**  
**CA300001**

7440 Lincoln Way  
Garden Grove, CA 92841-1427

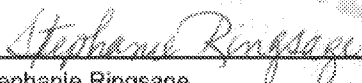
IS GRANTED APPROVAL BY ORELAP UNDER THE 2009 TNI STANDARDS, TO PERFORM  
ANALYSES ON ENVIRONMENTAL SAMPLES IN MATRICES AS LISTED BELOW :

<i>Air</i>	<i>Drinking Water</i>	<i>Non Potable Water</i>	<i>Solids and Chem. Waste</i>	<i>Tissue</i>
Chemistry		Chemistry	Chemistry	

AND AS RECORDED IN THE LIST OF APPROVED ANALYTES, METHODS, ANALYTICAL  
TECHNIQUES, AND FIELDS OF TESTING ISSUED CONCURRENTLY WITH THIS CERTIFICATE  
AND REVISED AS NECESSARY.

ACCREDITED STATUS DEPENDS ON SUCCESSFUL ONGOING PARTICIPATION IN THE  
PROGRAM AND CONTINUED COMPLIANCE WITH THE STANDARDS.

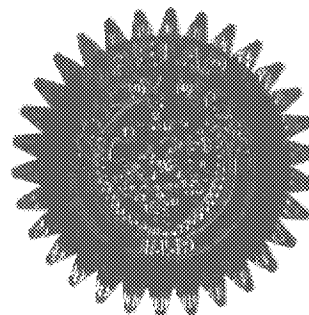
CUSTOMERS ARE URGED TO VERIFY THE LABORATORY'S CURRENT ACCREDITATION  
STATUS IN OREGON.

  
Stephanie Ringsage  
Oregon State Public Health Laboratory  
ORELAP Program Manager  
7202 NE Evergreen Parkway, Suite 100  
Hillsboro, OR 97124

EFFECTIVE DATE : 01/30/2020

EXPIRATION DATE : 01/29/2021

Certificate No : CA300001 - 013





## AIHA Laboratory Accreditation Programs, LLC

*acknowledges that*

### LA Testing

**520 Mission Street, South Pasadena, CA 91030**

**Laboratory ID: LAP-102814**

along with all premises from which key activities are performed, as listed above, has fulfilled the requirements of the AIHA Laboratory Accreditation Programs (AIHA-LAP), LLC accreditation to the ISO/IEC 17025:2005/2017 international standard, General Requirements for the Competence of Testing and Calibration Laboratories in the following:

#### LABORATORY ACCREDITATION PROGRAMS



**INDUSTRIAL HYGIENE**

Accreditation Expires: April 01, 2022

**ENVIRONMENTAL LEAD**

Accreditation Expires:

**ENVIRONMENTAL MICROBIOLOGY**

Accreditation Expires: April 01, 2022

**FOOD**

Accreditation Expires:

**UNIQUE SCOPES**

Accreditation Expires:

Specific Field(s) of Testing (FoT)/Method(s) within each Accreditation Program for which the above named laboratory maintains accreditation is outlined on the attached Scope of Accreditation. Continued accreditation is contingent upon successful on-going compliance with ISO/IEC 17025:2005/2017 and AIHA-LAP, LLC requirements. This certificate is not valid without the attached Scope of Accreditation. Please review the AIHA-LAP, LLC website ([www.aihaaccreditedlabs.org](http://www.aihaaccreditedlabs.org)) for the most current Scope.

Elizabeth Bair  
Chairperson, Analytical Accreditation Board

Cheryl O Morton  
Managing Director, AIHA Laboratory Accreditation Programs, LLC

Revision 17: 09/11/2018

Date Issued: 12/31/2019





## AIHA Laboratory Accreditation Programs, LLC

### SCOPE OF ACCREDITATION

#### LA Testing

520 Mission Street, South Pasadena, CA 91030

Laboratory ID: LAP-102814

Issue Date: 08/31/2018

The laboratory is approved for those specific field(s) of testing/methods listed in the table below. Clients are urged to verify the laboratory's current accreditation status for the particular field(s) of testing/Methods, since these can change due to proficiency status, suspension and/or withdrawal of accreditation.

#### Industrial Hygiene Laboratory Accreditation Program (IHLAP)

Initial Accreditation Date: 11/01/2003

IHLAP Scope Category	Field of Testing (FOT)	Technology sub-type/Detector	Published Reference Method/Title of In-house Method	Component, parameter or characteristic tested
Asbestos/Fiber Microscopy Core	Phase Contrast Microscopy (PCM)	-	NIOSH 7400	-

A complete listing of currently accredited IHLAP laboratories is available on the AIHA-LAP, LLC website at: <http://www.aihaaccreditedlabs.org>

United States Department of Commerce  
National Institute of Standards and Technology



**Certificate of Accreditation to ISO/IEC 17025:2005**

NVLAP LAB CODE: 200232-0

**LA Testing**  
South Pasadena, CA

*is accredited by the National Voluntary Laboratory Accreditation Program for specific services,  
listed on the Scope of Accreditation, for:*


**Asbestos Fiber Analysis**

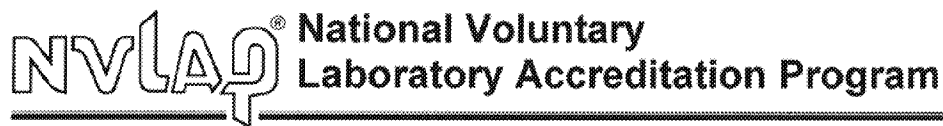
*This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2005.  
This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality  
management system (refer to joint ISO-ILAC-IAF Communique dated January 2009).*

2019-07-01 through 2020-06-30

*Effective Dates*



  
*For the National Voluntary Laboratory Accreditation Program*



## SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

### LA Testing

520 Mission Street  
South Pasadena, CA 91030  
Mr. Jerry Drapala Ph.D.  
Phone: (323) 254-9960 Fax: (323) 254-9982  
Email: [jdrapala@latesting.com](mailto:jdrapala@latesting.com)  
<http://www.latesting.com>

### ASBESTOS FIBER ANALYSIS

NVLAP LAB CODE 200232-0

#### Bulk Asbestos Analysis

<u>Code</u>	<u>Description</u>
18/A01	EPA -- 40 CFR Appendix E to Subpart E of Part 763, Interim Method of the Determination of Asbestos in Bulk Insulation Samples
18/A03	EPA 600/R-93/116: Method for the Determination of Asbestos in Bulk Building Materials

#### Airborne Asbestos Analysis

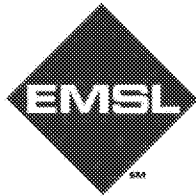
<u>Code</u>	<u>Description</u>
18/A02	U.S. EPA's "Interim Transmission Electron Microscopy Analytical Methods-Mandatory and Nonmandatory-and Mandatory Section to Determine Completion of Response Actions" as found in 40 CFR, Part 763, Subpart E, Appendix A.

A handwritten signature in black ink, reading "Dana S. Gorman".

For the National Voluntary Laboratory Accreditation Program

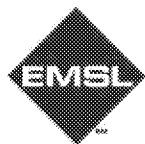
Effective 2019-07-01 through 2020-06-30

Page 1 of 1



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EMSL Analytical, Inc. Management



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

## EMSL Analytical S.O.P.

### Asbestos and Other Fibers by PCM 7400

*NIOSH 7400 Issue 3 – June 14, 2019*

*[ASB-SOP-300]*

#### 1.0 Method Description

##### 1.1 Applicable Matrix

This procedure is for the determination of fibers in air by phase contrast microscopy. Air samples are collected on 0.45 to 1.2  $\mu\text{m}$  mixed cellulose ester membrane (MCE) filters, using 25 mm cassettes with an electrically conductive cowl, at air flow rates of 0.5 to 16 L/min.

##### 1.2 Scope and Application

The procedures outlined in this SOP are based on the NIOSH 7400 method, and provide a concentration of optically visible airborne fibers. This method is primarily used for air monitoring during asbestos related activities, but since PCM does not differentiate between asbestos and other fibers, results can only be reported in overall fiber concentrations. This method may be used in conjunction with transmission electron microscopy (NIOSH 7402 method) to estimate the asbestos fiber concentration of a sample.

##### 1.3 Summary of Method

The sample is collected utilizing a MCE air cassette. The filter is cleared and fixed on a microscope slide by either an acetone vapor, or Dimethylformamide (DMF). The cleared filter preps are then mounted with a cover slip, using either Triacetin or Euparal. Fibers are counted at a magnification of 400X, using a positive phase contrast microscope.

##### 1.4 Detection Limit

The limit of detection for this method is 7 fibers/ $\text{mm}^2$  (or 5.5 fibers/100 fields) as defined by the published NIOSH 7400 method.

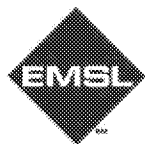
#### 2.0 Interferences

Interferences for this method include, but are not limited to the following:

- 2.1 High levels of non-fibrous dust particles may obscure fibers and indirectly increase the detection limit of a sample.
- 2.2 Some types of airborne spores may appear fiber-like and cause artificially high readings.
- 2.3 Chain-like particles may be mistaken for fibers.
- 2.4 When this method is used for monitoring a specific fiber type, the presence of any other airborne fibers may interfere, since all particles meeting the counting criteria are included in the result.

#### 3.0 Definitions

- 3.1 **Aspect Ratio:** The ratio of length to width of an object
- 3.2 **Coefficient of Variance:** Also known as Relative Standard Deviation ( $S_r$ ), this is a measure of the expected difference between a number of repeat analyses
- 3.3 **Fiber:** An elongated particle, with an aspect ratio of at least 3:1, and a minimum length of  $>5\mu\text{m}$



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

- 3.4 **MCE Filter:** Mixed Cellulose Ester is a type of filter typically used for PCM analysis.
- 3.5 **NIOSH:** National Institute of Occupational Safety and Health
- 3.6 **PCM:** Phase Contrast Microscopy is a form of microscopy that enhances differences in the refractive index of particles, adding to their contrast in the microscope's field of view.
- 3.7 **STEL:** Short Term Exposure Limit defined by the American Conference of Governmental Industrial Hygienists (ACGIH) as the concentration to which workers can be exposed for a short period of time (typically 15 minutes), as long as the 8 hour Time Weighted Average (TWA) is not exceeded. Time Weighted Average calculations and determination of STEL or TWA exceedance is an industrial hygiene function, and not a lab responsibility. EMSL provides TWA calculations for informational purposes only at the request of our clients.
- 3.8 **TWA:** Time Weighted Average is a calculated estimation of the effective fiber concentration a person would be exposed to if all exposure were distributed evenly over 8 hours (a normal work day).
- 3.9 **Walton-Beckett Graticule:** An inscribed circle in the eyepiece of the microscope ocular. The graticule is a circle of known area that defines the field of view for analysis, and provides various measuring aids to help the analyst evaluate fibers for aspect ratio and size.

## 4.0 Safety

All personnel performing preparation and/or analysis of samples must be familiar with the EMSL Chemical Hygiene and Safety Plan, which can be found on EMSL's eLink site.

### 4.1 Asbestos

- 4.1.1 All samples must be treated as potentially asbestos containing. Prudent measures must be taken to prevent any possible airborne asbestos fiber release from occurring during sample handling.
- 4.1.2 Any filter handling performed prior to the filter clearing step shall be performed under the safety hood.
- 4.1.3 All safety hoods shall be capable of flow rates  $\geq 75$  lfm (lineal feet per minute).

### 4.2 Acetone

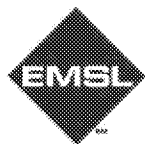
- 4.2.1 Keep away from heat, sparks, and flame.
- 4.2.2 Avoid breathing vapors; use with adequate ventilation.
- 4.2.3 Avoid contact with eyes.
- 4.2.4 Prevent prolonged or repeated contact with skin.

### 4.3 Dimethylformamide (DMF)

- 4.3.1 DMF is toxic by inhalation and through absorption by the skin.
- 4.3.2 When warming the slide prep with DMF for clearing, it is best to have the drying oven or warming plate within a fume exhaust hood, or ducted to the outside.

## 5.0 Equipment and Supplies

- 5.1 Acetone vaporizer or HotBlock™
- 5.2 Cover slips, 1.5 thickness
- 5.3 Relocatable gridded cover slip (optional)
- 5.4 Forceps
- 5.5 Kimwipes
- 5.6 Mechanical counter / tally counter
- 5.7 Micropipette(s) capable of dispensing 5  $\mu$ l



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

- 5.8 Disposable pipette or dropper bottle
- 5.9 Microscope slides, 3x1 inch
- 5.10 Phase contrast microscope with green or blue filter
- 5.11 8x-10x oculars
- 5.12 40x-45x objective with a numerical aperture of 0.65 to 0.75
- 5.13 #10 scalpel with a curved blade
- 5.14 Syringe capable of dispensing 100-500  $\mu$ l
- 5.15 Telescoping ocular or Bertrand lens
- 5.16 Walton Beckett graticule, type G-22
- 5.17 HEPA filtered safety hood

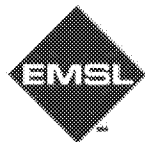
## 6.0 Reagents and Standards

All reagents shall be of recognized analytical grade, or better:

- 6.1 Acetone
- 6.2 Triacetin
- 6.3 Dimethylformamide (DMF); *optional*
- 6.4 Glacial Acetic Acid; *optional*
- 6.5 Euparal; *optional*
- 6.6 HSE/NPL or HSE/ULO red, green or yellow test slide with an invisible set of lines
- 6.7 Stage micrometer traceable to NIST (verified from outside agency once)
- 6.8 Past proficiency samples
- 6.9 Consensus standards

## 7.0 Sample Collection, Preservation, Shipment and Storage

- 7.1 Samples are collected on 0.45 to 1.2  $\mu$ m pore size 25mm MCE filters in sample cassettes with a conductive cowl.
- 7.2 Air flow shall be between 0.5 and 16 LPM (liters per minute). It is recommended the sampling flow rate not exceed 10 LPM.
- 7.3 Sample volume shall be adjusted to give 100 to 1300 fibers/ $\text{mm}^2$ , where possible.
- 7.4 No sample preservation is needed, and samples can be stored indefinitely prior to analysis.
- 7.5 Samples are best transported to the lab by hand. When mailing, try to package samples carefully to minimize disturbance and possible dislocation of particulate from the filter surface. Use packing materials that will minimize static charge. It is recommended to avoid the use of "foam peanut" packing material.
- 7.6 All air cassettes must be retained in an easily retrievable manner for a minimum of 60 days.
- 7.7 PCM slide preps may be discarded only after PCM QC is complete and evaluated satisfactorily.
- 7.8 Slide standards (stage micrometer, HSE/NPL or HSE/ULO test slides) may sometimes need to be shipped between laboratories. When this is necessary, use the following procedure:
  - 7.8.1 The slide in its storage enclosure shall be taped shut.
  - 7.8.2 The container shall then be wrapped in bubble wrap to protect the slide and container.
  - 7.8.3 If the slide is to be transported by hand, no further steps are necessary.
  - 7.8.4 If the slide is to be shipped by 3<sup>rd</sup> party courier, enclose the wrapped slide in an appropriate transport container (envelope or box) and use more packing material, as necessary, to avoid jostling and damage during transport.



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

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- 7.8.5** Seal the container and submit to 3<sup>rd</sup> party transport agency, including completion of custody forms.

## 8.0 Calibration and Standardization

Each major component of the method is calibrated and/or standardized including the analyst. Examples follow:

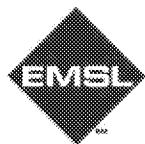
- 8.1** Sample collection vacuum pumps are calibrated at the beginning and end of a sampling event (with the sample cassette in line) using a rotameter.
- 8.2** The rotameter in turn needs to be calibrated to a primary standard periodically. (Rotameter use and calibration is the responsibility of the sample collection entity.)
- 8.3** All PCM microscope calibrations are based on frequency and are considered per analyst / per microscope.

**Note:** EMSL has various models of PCM microscopes in use; not all microscopes are as adjustable as others. To that extent, certain alignment steps may not be completely adjustable on all models. As long as the end user performs the steps that are possible on the microscope in question, and the proper alignment is verified, then the microscope alignment is considered to be completed.

The PCM microscope calibrations are recorded in iL@B to ensure:

- 8.3.1** The inter-pupillary distance is adjusted for the user's eyes. The focus of the Walton Beckett graticule is verified and adjusted for the user's vision.
- 8.3.2** If possible, adjust the light source for even illumination across the field of view. Frequency is daily, or on next use per analyst / microscope combination.
- 8.3.3** Center and focus the field iris (if possible) so it is open only enough to fully illuminate the field of view. Adjust the height of the sub-stage condenser to sharpen the image of the field iris. Frequency is daily, or on next use per analyst / microscope combination.
- 8.3.4** Using a telescoping ocular or Bertrand lens, focus the image of the rings (both sub-stage and in the 40x phase shift objective lens). If the rings do not form concentric circles, adjust the condenser adjustment screws to bring the rings into alignment. Frequency is daily, or on next use per analyst / microscope combination.
- 8.3.5** Check the shift detection limit using the HSE/NPL (**Mark II**) or HSE/ULO (**Mark III**) test slide. Each of the slides consists of seven (7) sets of increasingly narrowing diameter inscribed lines. Each class of slide has different line resolutions; the lines able to be seen in each are listed below. Frequency is weekly per analyst / microscope combination.
  - 8.3.5.1** HSE/NPL (Mark II): The microscope shall be able to completely resolve the first 3 sets of lines (1-3); the next two sets of lines (4-5) shall be completely or partially visible. The microscope shall not be able to see the last two sets (6-7) of lines at all; they shall be invisible.
  - 8.3.5.2** HSE/ULO Red (Mark III Red): The microscope shall be able to completely resolve the first 4 sets of lines (1- 4); the set of lines (5) shall be partially visible. The microscope shall not be able to see the last two sets (6-7) of lines at all; they shall be invisible.
  - 8.3.5.3** HSE/ULO Green (Mark III Green): The microscope shall be able to completely resolve the first 5 sets of lines (1-5); the set of lines (6) shall be partially visible. The





microscope shall not be able to see the last set (7) of lines at all; they shall be invisible.

**8.3.5.4 HSE/ULO Yellow (Mark III Yellow):** This slide does not contain a set of invisible lines and is therefore not acceptable for this calibration, and shall not be used.

**8.3.6** The area of the Walton Beckett graticule shall be measured to ensure the correct field area is used for analytical calculations. The graticule diameter is measured to the nearest division of the stage micrometer (the goal is for the final measurement to be expressed to the nearest  $\mu\text{m}$ ). The graticule area is calculated using the formula for the area of a circle ( $\pi r^2$ ) where  $r$  is the radius of the graticule field. EMSL will only use a Walton Beckett graticule that is exactly 100 $\mu\text{m}$ , and produce a field area of 0.00785 $\text{mm}^2$ , which is used in all report calculations. If the graticule area is different than this stated value, a new graticule shall be procured that will meet this requirement. Frequency is monthly per analyst / microscope combination; for labs meeting the TNI Standard (NELAC), the frequency shall be daily per analyst / microscope combination.

**8.4** Mechanical counter accuracy is documented by counting to a minimum of 100 while clicking the counter with each count. The clicker shall read 100 on the 100<sup>th</sup> count. Frequency is monthly.

**8.5** Analyst precision and accuracy frequency is detailed in Quality Control Section 13.0.

**8.5.1** A reference slide is submitted to each analyst on a daily basis. This slide shall be submitted blindly so the analyst does not know the identity of the sample. The reference slide library shall consist of old proficiency, round robin and real world (client) samples, where the target concentration and acceptance limits have been established and are known.

**8.5.2** Measuring Accuracy: To measure and judge accuracy, the analysis must fall within the acceptable limits before analysis may proceed. If the sample falls out of the limits:

**8.5.2.1** A Corrective Action Response (CAR) must be initiated.

**8.5.2.2** Another reference slide in the same fiber concentration range must be analyzed, and results fall within acceptable range before client sample analysis may proceed.

**8.5.2.3** The CAR does not need to be closed before analysis may proceed.

**8.5.3** Measuring Precision: Precision is calculated by use of a spreadsheet log of all analyses of each reference slide per analyst.

**8.5.3.1** The most recent 20 analyses of each slide are considered for this measurement and the pooled SD and mean are calculated; the coefficient of variance (CV) or relative standard deviation ( $S_r$ ) is then determined. This is detailed in ASB-SOP-1 Quality Control.

**8.5.3.2** CV ( $S_r$ ) results of  $>0.45$  shall be considered suspect, and a CAR initiated.

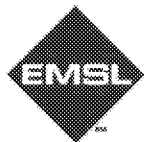
**8.5.4** Traceability: All reagents must be recorded in the GEN-FM-501 Stock Standard and Reagent Log. Primary preparation reagents Acetone and Triacetin are recorded in IL@B.

**8.5.5** The use of relocatable gridded cover slips with Euparal mounted samples and reference samples may be used for training.

## 9.0 Procedure

### 9.1 Sample Receipt

**9.1.1** Upon receipt of samples, ensure the sample information on the Chain of Custody (COC) matches the information on the samples and other paperwork. Any discrepancies must be resolved before proceeding.



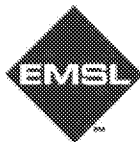
ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

- 9.1.2** If the samples are not accompanied by a COC, then one is initiated at time of login. Have the client fill out the necessary information completely.
- 9.1.3** Information required on the Chain of Custody includes:
- 9.1.3.1** Client name, address, telephone number, contact person, fax number
  - 9.1.3.2** Project number/ name, state where samples were taken
  - 9.1.3.3** Number of samples submitted and sample IDs
  - 9.1.3.4** Type of analysis requested
  - 9.1.3.5** Sample volumes or areas, if applicable
  - 9.1.3.6** Specific turn-around time (TAT) requested ("RUSH" is not acceptable)
  - 9.1.3.7** A date and signature of the person relinquishing the samples
  - 9.1.3.8** All samples MUST be accounted for with the proper sample IDs.
  - 9.1.3.9** All samples MUST be sealed, properly bagged and undamaged.
- 9.1.4** All samples must be clocked in at the time of receipt and signed and dated by an EMSL employee. If the lab does not have a clock for sample receipt, the receiving employee shall record the time of receipt.
- 9.1.5** Check to see if the samples match the COC and if the cassettes are open, damaged, or contaminated. If the samples are damaged or if the COC does not match, notify the client.
- 9.2 Sample Login**
- If all of the above criteria for sample receiving are met, the samples can be logged in to EMSL's LIMS (Sample Master) as per GEN-SOP-702 Sample Receiving and Chain of Custody.
- 9.2.1** This process will assign a unique EMSL order number for the project, as well as unique lab sample IDs.
  - 9.2.2** Sample Master will create and print an internal chain of custody.
  - 9.2.3** Care shall be taken for blank correction purposes to identify samples and field blanks from the same batch of samples accordingly. SMXP allows for a Sample Type field to be completed during login that "links" certain field blank samples to associated sample IDs.
  - 9.2.4** All samples in a given "set" shall be logged in with "Customer Set X," where "x" is a set of samples from 1 to 7.
  - 9.2.5** Associated field blanks for each set shall be logged in as "Field Blank X," where "x" is a set of field blanks from 1 to 7 that corresponds to the "Customer Set" of samples in step 9.2.4, above.
  - 9.2.6** SMXP will now link the samples and blanks and perform a blank correction (following the procedures outlined in Sections 9.4.15 or 9.5.15 for A or B rules, respectively) automatically on the associated filters.
- 9.3 Sample Preparation (although optional alternative preparation methods are outlined in this SOP and the NIOSH 7400 method, EMSL will continue to use the Acetone and Triacetin procedure as the primary prep method)**
- 9.3.1** In a safety hood with the blower on, place the samples in order corresponding to the COC. Cut the outer band (if present) on the cassettes with a scalpel or straight razor.
  - 9.3.2** Lay out a clean 3x1 microscope slide. A slide can accommodate 2 or 3 sample wedges, depending on the person prepping.
  - 9.3.3** Clean the tools used to handle and cut the samples prior to handling the first sample filter, and then between each sample. Do not use the same blade used to cut the band to cut the wedge in the next step.



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

- 9.3.4** Cut a wedge of the filter using a curved blade in a rocking motion, and use clean forceps to place on the slide. Care must be taken to minimize disturbance of the fibers; therefore, handle the sample filters around the edge and on the stamped cowl section on the filter perimeter. Filters can be cut while in the cap or placed on a clean microscope slide to be cut.
- 9.3.5** Place the cut wedges in the corresponding sample order (two to three per slide), starting at the top (if laid out vertically) or the left side (if laid out horizontally) of each slide. Place 2-3 wedges on each slide.
- 9.3.6** When samples have been cut and their wedges placed on the slide, maximizing the distance between wedges, collapse the filters using the acetone vapor generator.

**9.3.6.1 Procedure (Acetone and Triacetin):**

**9.3.6.1.1** Draw fresh acetone into a syringe; insert the syringe into the top of the acetone vaporizer or hot block.

**9.3.6.1.2** Insert the slide into the area where the acetone vapors are exhausted, centered on the first wedge to be cleared. Slightly depress the plunger of the syringe; the amount of acetone shall be just enough to clear the first wedge on the slide, approximately 250  $\mu$ l. Move the slide to the next wedge and continue the process until all wedges on the slide are clear.

**Note:** Avoid excess acetone or acetone used when vaporizer is not at proper temperature, which may cause materials to be washed off the surface of the filter.

**9.3.6.1.3** One drop of (approximately 3.0-3.5  $\mu$ l) of triacetin per cleared filter wedge, within 30 seconds of acetone clearing, may be applied using a syringe, disposable pipette, 5 $\mu$ l pipette, or dropper bottle. It is important not to use too much triacetin, or to touch the sample with the pipette tip. An excess amount of triacetin may cause particulate and/or fibers to migrate on the filter, and in extreme cases, may cause the cover slip to float.

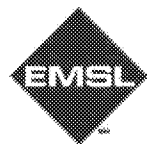
**9.3.6.1.4** Carefully place a clean cover slip on each sample. Do not press down on the cover slip, as this will foster fiber migration on the filter.

**9.3.6.1.5** Place the prepped slide on a slide warmer or hot surface (approximately 50°C) of the acetone vaporizer to complete clearing. When the triacetin is applied to samples after the acetone-cleared sample has been allowed to sit momentarily, the slide preparation may become cloudy. This step is needed to ensure a clear prep, and may not be necessary if triacetin and coverslip are applied quickly after acetone clearing. Continue until all samples are prepped.

**9.3.6.2 Optional Clearing Method using DMF/Acetic Acid:**

**9.3.6.2.1** DMF prep must be performed in a fume hood, and may be useful when samples cannot be cleared sufficiently by acetone prep.

**9.3.6.2.2** Make a mixture of 1.4ml (35% volume) of DMF, 0.6ml (15% volume) glacial acetic acid, and 2ml (50% volume) of distilled water. This solution must be replaced weekly.



**9.3.6.2.3** Place filter wedge(s) in their proper position on a glass microscope slide.

**9.3.6.2.4** Place  $20 \pm 5$   $\mu$ l of DMF solution in small drops along the edge of the filter(s), allowing the solution to wick onto the filter.

**9.3.6.2.5** Place slide in drying oven or on hotplate at  $60 (+2/-5)^{\circ}\text{C}$  for 30 minutes.

**9.3.6.2.6** Place one drop (approximately 3.0-3.5  $\mu$ l) of triacetin on each cleared filter wedge. This can be done using a syringe, disposable pipette, 5 $\mu$ l pipette, or dropper bottle. It is important not to use too much triacetin, or to touch the sample with the tip. An excess amount of triacetin may cause particulate and/or fibers to migrate on the filter, and in extreme cases, may cause the cover slip to float.

**9.3.6.2.7** Carefully place a clean cover slip on each sample. Do not press down on the cover slip, as this will foster fiber migration on the filter.

**9.3.6.3 Optional Euparal Mount (after clearing, to make a more stable, permanent mount)**

**9.3.6.3.1** Add 1 drop of Euparal solution in the middle of the cleared wedge, and 1 drop to the center of the cover slip. Gently lower cover slip onto slide at a slight angle to avoid bubbles.

**9.3.6.3.2** Warm slide at  $60 (+2/-5)^{\circ}\text{C}$  for 1 hour.

**9.3.7** Optional: Outline the filter segment just inside the segment with a 'Sharpie' marker. Markings can be made on the bottom of the slide or on the coverslip. If marking the cover slip is preferred, it must be done lightly and carefully to avoid pressure applied to sample causing disturbance of fibers.

**9.3.8** With a permanent marker (such as a 'Sharpie'), write the sample numbers on the slide. This may be done prior to sample prep, when the slides are being laid out in the hood. Care is needed to not "wash" the marking away during the sample clearing process with acetone vapors. In practice this is possible, but difficult.

**9.3.9** If the samples cannot be read within 24 hours of prep, each sample cover slip must be sealed with clear nail polish to preserve the prep.

**9.3.10** Initial analyses and QC activities must be completed prior to the release of results to the client.

**9.4 Sample Analysis "A" Rules**

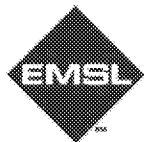
Under normal circumstances, analysis of samples via NIOSH 7400 is performed by the "A" counting rules. At the client's request alternate "B" counting rules are available (Section 9.5 of this procedure). In the absence of any contrary direction by the client, the "A" rules will be used for analysis of PCM samples via NIOSH 7400.

**9.4.1** Place the first slide of the sample set to be analyzed on the microscope stage. Adjust the sample translators to bring the first sample to be analyzed into the microscope's field of view. It may be helpful to view the slide at a low magnification (40x) to find and center the samples.

**9.4.2** With the first sample in the field of view, and at a magnification of 400x, focus the sample, taking care not to allow the 40x objective to touch the sample cover slip.

**9.4.3** Once the filter is focused, observe the filter.

**9.4.3.1** If the filter is judged to be loaded with >50 particulate, reject the filter as being too heavily loaded and report as "Overloaded."



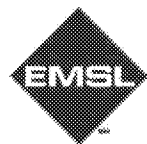
ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

- 9.4.3.2** If the filter contains a sample loading that is uneven, reject the filter and report it as "Non-Uniform Distribution."
- 9.4.4** If the filter is acceptable for analysis, move to the cut tip of the filter. Using one stage adjustment control, move the slide to a random field of view, in a direction away from the cut tip and toward the outer edge of the filter.
- 9.4.5** Focus on the field of view, including over and under focusing to see all fibers in the field. If an agglomerate or bubble covers 1/4 of the field or more, reject the field and move on.
- 9.4.6** Fibers as defined by this method are:
- 9.4.6.1** fibers  $>5\mu\text{m}$  in length and
  - 9.4.6.2** having an aspect ratio  $\geq 3:1$
- 9.4.7** Count fibers that are completely within the graticule field as one (1) fiber.
- 9.4.8** Count fibers that cross the graticule boundary as follows:
- 9.4.8.1** Count as one half ( $\frac{1}{2}$ ) fiber any fiber with only one end lying within the graticule field.
  - 9.4.8.2** Do not count any fiber that crosses the boundary more than once.
- 9.4.9** Do not count fibers that lie completely outside the graticule field.
- 9.4.10** Count bundles as one (1) fiber unless both ends of individual fibers can be observed; if so, count the fibers separately.
- 9.4.11** Count fibers protruding from matrix material, providing at least  $5\mu\text{m}$  of the fiber is visible, and the protrusion has at least a 3:1 aspect ratio.
- 9.4.12** Once all fibers within the graticule have been counted, move to the next field of view in the same direction. The analyst shall move along a radial line toward the outer edge of the filter when selecting fields of view. Each new field of view shall be sufficiently far away from the previous field of view so the Walton Beckett graticule is not superimposed on areas in the previous field of view.
- 9.4.13** Once the edge of the filter has been reached, shift up or over and continue counting in the opposite direction.
- 9.4.14** Continue counting until:
- 9.4.14.1** at least 100 fields have been counted
  - 9.4.14.2** at least 100 fibers have been counted in a minimum of 20 fields
  - 9.4.14.3** always finish counting the final field of the analysis; never stop counting mid-field
- 9.4.15** Information is recorded on EMSL's PCM worksheet, or in EMSL's Direct Data entry database interface "iLab." For information on the use of "iLab," please see EMSL's Asbestos DDE Manual. Regardless of the method of recording used, all information detailed below is required to be recorded:
- 9.4.15.1** the number of fibers counted
  - 9.4.15.2** the number of fields analyzed
  - 9.4.15.3** a check in the "Overloaded" column if the filter was overloaded
  - 9.4.15.4** Any comments that may be pertinent for the sample. This could include if the sample had an uneven loading, if the filter was wet and required drying, the sample volume, or any other comment the analyst believes is important enough to record.
- 9.4.16** Repeat the previous steps in this section until all samples have been analyzed. Once all samples and field blanks in the set have been completed, the samples shall be blank corrected. To blank correct:



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

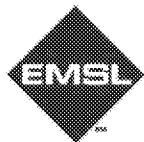
- 9.4.16.1** Total the number of fibers counted on the field blanks for each batch of samples. It is possible one EMSL order number may contain more than one batch of samples and multiple sets of field blanks. It is important only the field blanks for each batch be averaged and applied to the batch of samples they apply to, as described below.
- 9.4.16.2** If a field blank has been noted for "Possible Contamination" (see 14.1.4 below), it shall not be included in the average field blank count.
- 9.4.16.3** Divide the total fibers on the field blanks by the number of field blanks in the batch of samples of interest. Record this on the bottom of the analytical sheet in the spot for "Average Fibers per 100 fields for the Field Blanks."
- 9.4.16.4** Subtract the average number of fibers for the field blanks from each sample the field blanks represent. If the sample to be blank corrected consisted of less than 100 fields, the field blank fiber average must be extrapolated before subtraction. This process is described in Section 10.3 of Calculations.
- 9.4.16.5** Record this number in the "# Blank Corrected Fibers" column for each sample. This is not necessary for samples processed using iLab, as "iLab" internally calculates blank correction.
  - 9.4.16.5.1** Do not blank correct field blanks.
  - 9.4.16.5.2** If the results of this calculation would produce a negative number, record zero (0).
- 9.4.17** After all blank correction has been completed, complete the bottom of the analytical bench sheet by (handled internally if using "iLab"):
  - 9.4.17.1** signing the analyst's signature
  - 9.4.17.2** filling in the date of analysis
  - 9.4.17.3** filling in the scope ID

## 9.5 Sample Analysis "B" Rules

**Note:** NYS ELAP does not include the B Rules as part of their scope of accreditation.

At the client's discretion and request, EMSL will analyze PCM samples by NIOSH 7400 "B" rules. This alternate set of counting rules has different criteria for fibers; the "B" rule's fiber criteria are listed in item 9.5.5 below. If a client requests "B" rules be used for analysis, the entire sample must be analyzed by "B" rules. It is not permissible to produce the analysis using both, or a hybrid of both, sets of rules. Unless the client specifies this alternate counting protocol, EMSL will use the "A" rules for routine analysis of samples via NIOSH 7400.

- 9.5.1** Place the first slide of the sample set to be analyzed on the microscope stage. Adjust the sample translators to bring the first sample to be analyzed into the microscope's field of view. It may be helpful to view the slide at a low magnification (40x) to find and center the samples.
- 9.5.2** With the first sample in the field of view, and at a magnification of 400x, focus the sample, taking care not to allow the 40x objective to touch the sample cover slip.
- 9.5.3** Once the filter is focused, observe the filter.
  - 9.5.3.1** If the filter is judged to be loaded with >50 particulate, reject the filter as being too heavily loaded and report as "Overloaded."
  - 9.5.3.2** If the filter contains a sample loading that is uneven, reject the filter and report it as "Non-Uniform Distribution."



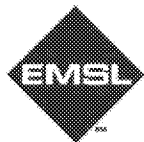
ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

- 9.5.4** If the filter is acceptable for analysis, move to the cut tip of the filter. Using one stage adjustment control, move the slide to a random field of view, in a direction away from the cut tip and toward the outer edge of the filter.
- 9.5.5** Focus on the field of view, including over and under focusing to see all fibers in the field. If an agglomerate or bubble covers 1/4 of the field or more, reject the field and move on.
- 9.5.6** Fibers as defined by this part of the method are:
- 9.5.6.1** fibers  $>5\mu\text{m}$  in length and
  - 9.5.6.2** fibers  $<3\mu\text{m}$  in width and
  - 9.5.6.3** having an aspect ratio  $\geq 5:1$
- 9.5.7** Count each fiber end that falls within the graticule field as one end.
- 9.5.8** Count split ends, as long as the split fiber segment meets the criteria in 9.5.5.
- 9.5.9** For fibers attached to particulate, count as follows:
- 9.5.9.1** Count visibly free ends that meet the criteria in 9.5.5 when they are attached to particulate, regardless of the size of the particle they are attached to.
  - 9.5.9.2** Count the end of a fiber obscured by another particle if the particle covering the fiber is  $<3\mu\text{m}$  in diameter.
  - 9.5.9.3** Count free ends of fibers attached to or emanating from large clumps or bundles, up to a maximum of 10 ends, provided each fiber segment counted meets the criteria in 9.5.5.
- 9.5.10** Do not count fibers that lie completely outside the graticule field.
- 9.5.11** Once all fibers within the graticule have been counted, move to the next field of view in the same direction. The analyst shall move along a radial line toward the outer edge of the filter when selecting fields of view. Each new field of view shall be sufficiently far away from the previous field of view, so the Walton Beckett graticule is not superimposed on areas in the previous field of view.
- 9.5.12** Once the edge of the filter has been reached, shift up or over and continue counting in the opposite direction.
- 9.5.13** Continue counting until:
- 9.5.13.1** at least 100 fields have been counted or
  - 9.5.13.2** at least 200 ends have been counted in a minimum of 20 fields
  - 9.5.13.3** always finish counting the final field of the analysis; never stop counting mid-field
- 9.5.14** Divide the number of ends by two (2) to calculate total fibers.
- 9.5.15** Record on the analytical bench sheet in the appropriate line corresponding to the sample that was analyzed:
- 9.5.15.1** the number of fibers counted
  - 9.5.15.2** the number of fields analyzed
  - 9.5.15.3** a check in the "Overloaded" column if the filter was overloaded
  - 9.5.15.4** Any comments that may be pertinent for the sample. This could include if the sample had an un-even loading, if the filter was wet and required drying, the sample volume, or any other comment the analyst believes is important enough to record.
- 9.5.16** Repeat the previous steps in this section until all samples have been analyzed. Once all samples and field blanks in the set have been completed, the samples shall be blank corrected. To blank correct:



ASB-SOP-300 PCM NIOSH 7400

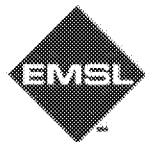
Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

- 9.5.16.1** Total the number of fibers counted on the field blanks for each batch of samples. It is possible one EMSL order number may contain more than one batch of samples and multiple sets of field blanks. It is important only the field blanks for each batch be averaged and applied to the batch of samples they apply to, as described below.
- 9.5.16.2** If a field blank has been noted for "Possible Contamination" (see 14.1.4 below), it shall not be included in the average field blank count.
- 9.5.16.3** Divide by the number of field blanks in the batch of samples of interest. Record this on the bottom of the analytical sheet in the spot for "Average Fibers per 100 fields for the Field Blanks."
- 9.5.16.4** Subtract the average number of fibers for the field blanks from each sample the field blanks represent. If the sample to be blank corrected consisted of less than 100 fields, the field blank fiber average must be extrapolated before subtraction. This process is described in Section 10.3 of Calculations.
- 9.5.16.5** Record this number in the "# Blank Corrected Fibers" column for each sample.
- 9.5.16.5.1** Do not blank correct field blanks.
- 9.5.16.5.2** If the results of this calculation would produce a negative number, record zero (0).
- 9.5.17** After all blank correction has been completed, complete the bottom of the analytical benchsheet by:
- 9.5.17.1** signing the analyst's signature
- 9.5.17.2** filling in the date of analysis
- 9.5.17.3** filling in the Scope ID
- 9.6 Procedures for Use of TWAs**
- 9.6.1** TWA samples are logged into Sample Master using the test "PCM w/TWA."
- 9.6.2** Ensure the client understands the calculation is based on an 8 hour exposure period. The final report is titled "...with 8 Hour Time Weighted Average."
- 9.6.3** Samples that are overloaded do not provide a viable analytical result and are not included in a TWA.
- 9.6.4** Sample Master calculates the TWA using the following criteria:
- 9.6.4.1** STEL (Short Term Exposure Limit) samples that overlap the sampling times of the associated personal samples are not included in the TWA.
- 9.6.4.2** For the TWA calculation, use the detection limit in f/cc for samples reported below the analytical detection limit.
- 9.6.4.3** If all samples are below their associated detection limits, report the final TWA as less than the calculated result.
- 9.6.4.4** Use the TWA calculation as found in Section 10.7 of Calculations, below.
- 9.6.5** Interpretation of these results is the responsibility of the industrial hygienist. TWA can be calculated using various exposure times as directed by the client. For times other than 8 hours, calculate the total exposure time in minutes (denominator). The final report must indicate what total time was used for the calculated result.
- 9.7 QC Recount Selection, Resubmission and Analysis**
- 9.7.1** Recount QC is selected randomly at a rate of 10% of real time analyses.
- 9.7.2** These recounts shall be resubmitted blindly to the original analyst, so they do not know the original sample results and preferably sample identity. Optimally, this will entail the selection





ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

and relabeling of QC recount samples by a second analyst or other lab personal (as per the method).

**9.7.3** QC samples to be analyzed are listed on a new blank PCM QC bench sheet, and resubmitted to the original analyst with the QC slides for analysis.

**9.7.4** Recounts are analyzed according to the procedures outlined in the analysis section of this SOP (9.4 for A rules; 9.5 for B rules).

**9.7.5** Recounts are recorded on an appropriate Blank QC bench sheet:

**9.7.5.1** Bench Sheets for Non-NYS Samples

### Fiber Count by Phase Contrast Microscopy (PCM), NIOSH 7400 Method, Issue 3, 6/14/19

Client:  
Address:

Logged:

TAT:

Date/Time Due:

Phone:  
Fax:  
Project:

Special Instructions

Order ID

Sample ID	Location	Sample Date	# Fibers	# Fields	# Blank Corrected	Overloaded	Comment

### Fiber Analysis of Air Samples via NIOSH 7400, Issue 3, 6/14/19 (with 8-Hour Time Weighted Average)

Includes OSHA Time-Weighted Average

Client:  
Address:

Logged:

TAT:

Date/Time Due:

Phone:  
Fax:  
Project:

Special Instructions

Order ID

Sample ID	Location	Sample Date	# Fibers	# Fields	Start	Stop	# Blank Corrected Fibers	Overloaded	Comment
					Rate				



ASB-SOP-300 PCM NIOSH 7400  
Revision 17  
Revision Date: July 22, 2019  
Effective Date: August 2, 2019

**9.7.5.2** In the case of NYSDOH ELAP certified labs, use a QC bench sheet that allows for recording the number of fibers in each field counted.

## NYS PCM QC Sheet

EMSL NYS PCM QC  
Revision 3  
May 2007

Analyst: \_\_\_\_\_

Scope : \_\_\_\_\_

Date: \_\_\_\_\_

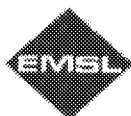
Date: \_\_\_\_\_

Lab #:						QC #:					
Orig	F/F					F/mm2					
QC:	F/F					F/mm2					
	1	2	3	4	5	6	7	8	9	10	
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											

Lab #:						QC #:					
Orig	F/F					F/mm2					
QC:	F/F					F/mm2					
	1	2	3	4	5	6	7	8	9	10	
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											

**9.7.5.3** Older style PCM QC recording tables shall no longer be used for PCM QC.

EMSL PCM QC Analysis Benchsheet  
Revision 1  
Effective Date: October 25, 2007

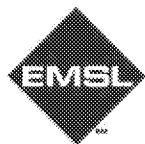


## PCM QC ANALYSIS BENCHSHEET

Month/Year: \_\_\_\_\_ Scope: \_\_\_\_\_

Date	Bill Number	Sample Number	Analysis	First Count fibers/fields	Second Count fibers/fields

**9.7.6** After QC analysis is complete, both original and QC results are entered into the PCM QC spreadsheet, as detailed in ASB-SOP-1 Asbestos Quality Control.



## 10.0 Calculations

When calculating fiber density and concentrations, if the fiber count is less than 5.5 fibers (the detection limit) use 5.5 fibers in place of the actual fiber count.

For all calculations:

- E - fiber density of filter in fibers / mm<sup>2</sup>
- C - fiber concentration of air sampled fibers / cc
- F - fibers counted during analysis
- B - average fiber count per field blank
- B<sub>corr</sub> - average fiber count per field blank corrected for samples where <100 field are counted
- F<sub>bc</sub> - blank corrected fiber count
- UCL - upper 95% confidence limit of fiber count
- A<sub>f</sub> - area of one (1) Walton Beckett graticule field (0.00785mm<sup>2</sup>)
- # - number of graticule fields analyzed in sample
- EFA - effective filter area of sample filter
- Vol - volume of air sampled in liters (L)
- CV - coefficient of variance

### 10.1 Filter Density

$$E = \frac{F_{bc}}{A_f \times \#} \quad E = \frac{12.5}{0.00785 \times 100} \quad E = 15.9 \frac{f}{mm^2}$$

### 10.2 Sample (Air) Concentration

$$C = \frac{EFA \times F_{bc}}{A_f \times \# \times 1000 \times Vol} \quad C = \frac{385 \times 12.5}{0.00785 \times 100 \times 1000 \times 1200} \quad C = 0.0051 \frac{f}{cc}$$

### 10.3 Fiber Count Blank Correction: Blank correction is provided in EMSL's reporting system.

**10.3.1** For fibers counts that end with 100 graticule fields analyzed:

$$F_{bc} = F - B \quad F_{bc} = 13 - 0.5 \quad F_{bc} = 12.5 \text{ fibers}$$

**10.3.2** For fiber counts that end with fewer than 100 graticule fields:

$$B_{corr} = \frac{\#}{100} \times B \quad B_{corr} = \frac{57}{100} \times 1.5 \quad B_{corr} = 0.855$$

Then

$$F_{bc} = F - B_{corr} \quad F_{bc} = 101 - 0.855 \quad F_{bc} = 100.15 \text{ fibers}$$

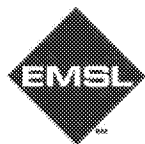
### 10.4 Upper Confidence Limit

The upper confidence limit is calculated for the fiber count. This result can then be substituted into the formulas for fiber density and/or sample concentration to calculate the UCL in either f/mm<sup>2</sup> or f/cc.

$$UCL = \frac{2 \times F_{bc} + [(2.25 + 2 \times F_{bc})^2 - 4(1 - 2.25 \times CV^2)F_{bc}^2]^{1/2}}{2(1 - 2.25 \times 0.25^2)}$$

$$UCL = \frac{2 \times 12.5 + 2.25 + [(2.25 + 2 \times 12.5)^2 - 4(1 - 2.25 \times 0.25^2)12.5^2]^{1/2}}{2(1 - 2.25 \times 0.25^2)}$$

$$UCL = 24.19 \text{ fibers}$$



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

### 10.5 QC Recount Acceptance / Rejection

Where E1 is the first or original result (in f/mm<sup>2</sup>), and E2 is the second or QC result. The test fails when the equation is true.

$$|\sqrt{E_1} - \sqrt{E_2}| > 2.77 \times \left( \frac{\sqrt{E_1} + \sqrt{E_2}}{2} \right) \times \frac{CV}{2}$$

$$|\sqrt{15.9} - \sqrt{25.5}| > 2.77 \times \left( \frac{\sqrt{15.9} + \sqrt{25.5}}{2} \right) \times \frac{0.18}{2}$$

1.08 > 1.13 is false, so the analysis passes

### 10.6 TWA (Time Weighted Average)

Where C<sub>i</sub> and T<sub>i</sub> are the concentrations (in f/cc) and time (in minutes) of a particular sample, up to and including the n<sup>th</sup> sample:

$$TWA = \frac{(C_1 \times T_1) + (C_2 \times T_2) + \dots + (C_n \times T_n)}{480}$$

$$TWA = \frac{(0.1 \times 60) + (0.02 \times 210)}{480}$$

$$TWA = 0.021 \frac{f}{cc}$$

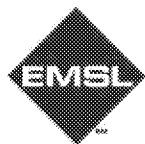
### 11.0 Reporting

The following items are included in the final report (on EMSL letterhead) to the client:

- 11.1 Fiber concentration in fibers/mm<sup>2</sup>
- 11.2 Fiber concentration in fibers/cc (if volume is supplied)
- 11.3 Detection limit of the analysis in fibers/cc
- 11.4 Number of fibers counted
- 11.5 Number of graticule fields analyzed
- 11.6 The date the sample was taken (if supplied)
- 11.7 Any notes pertaining to the sample
- 11.8 Volume of air collected by the client (if supplied)
- 11.9 Location of the sample in the field (if supplied)
- 11.10 Client and lab sample numbers
- 11.11 Client identification and contact information
- 11.12 EMSL Order ID
- 11.13 Client Project information (if supplied)
- 11.14 Sampling (if supplied), analysis and report date
- 11.15 Signature of Lab Manager
- 11.16 Report comments
- 11.17 Lab accreditations
- 11.18 The following disclaimers:

11.18.1 "Inter-laboratory Sr values: 5-20 fibers = 0.XX, 21-50 fibers = 0.XX, 51-100 fibers = 0.XX."

**Note:** These numbers are generated in the laboratory, and updated annually on all applicable report templates by the QA department.



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

**11.18.2** "Inter-laboratory  $S_r$  value = 0.XX."

**Note:** This number is generated by the QA department utilizing round robin data, and is updated annually on all applicable report templates by the QA department.

**11.18.3** "The laboratory is not responsible for the data in fibers/cc, which is dependent on volume collected by non-laboratory personnel."

**11.18.4** "This report relates only to the samples reported above. The report may not be reproduced, except in full, without written approval by EMSL."

**11.18.5** If results fall outside density range of 1300 f/mm<sup>2</sup> for the method, report disclaimer is as follows, "Fiber counts outside the recommended fiber density range of the method (100-1300 f/mm<sup>2</sup>) have greater than optimal variability, and are probably biased."

**11.18.6** If the sample contains a particulate loading of ≥50%, but the client requests the sample be analyzed anyway (if possible), the sample result shall be reported with a comment stating "Sample Overloaded, analyzed at client's request. Possibly Biased."

**11.19** For reports that determine TWA (time weighted average) the following are also reported:

**11.19.1** Activity instead of sample location

**11.19.2** Start and stop time of samples

**11.19.3** Flow rate of samples

**11.19.4** Notes/comments relating to whether the sample is included in the TWA or not

**11.19.5** TWAs broken down by collector and day collected

**11.20** Reporting Uncertainty to Clients

**11.20.1** Refer to the Excel spreadsheet **GEN-FM-302 Uncertainty – Asbestos**.

**11.20.2** The concentration at the upper and lower confidence limits will be calculated using the calculations in the NIOSH 7400 method (page 14).

$$UCL = \frac{2X + 2.25 + [(2.25 + 2X)^2 - 4(1 - 2.25 S_r^2)X^2]^{1/2}}{2(1 - 2.25 S_r^2)}$$

$$LCL = \frac{2X + 4 - [(4 + 2X)^2 - 4(1 - 4 S_r^2)X^2]^{1/2}}{2(1 - 4 S_r^2)}$$

Where:

$S_r$  = subjective inter-laboratory relative standard deviation, which is close to the total inter-laboratory  $S_r$  when approximately 100 fibers are counted

X = total fiber counted on sample

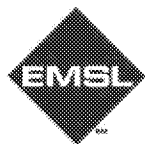
LCL = lower 95% confidence limit

UCL = upper 95% confidence limit

**Note:** The range between these two limits represents 90% of the total range.

**11.20.3** Results can be reported with a stated value of uncertainty when requested by the customer or accreditation agency. NIOSH 7400 is a well-established method which specifies the presentation of uncertainty.

**11.20.4** If requested by the client, this uncertainty is reported as the (+/-) % value without application to a specific result.



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

## 12.0 Method Performance

Method performance data can be found in the NIOSH Method 7400 - Asbestos and Other Fibers by PCM.

### 12.1 MDL

The method detection limit is stated as 7 fibers / mm<sup>2</sup>. This translates to 5.5 fibers per 100 fields using a graticule (field) area of 0.00785mm<sup>2</sup>.

### 12.2 DOCs

Demonstrations of Capability (DOC) are required for each analytical method. For compliance w/ NYS ELAP, when analyst sample loads exceed 2 samples per hour, an initial DOC (iDOC) consisting of a timed analysis of 25-30 PT samples must be conducted. This DOC must be challenged quarterly, and documentation of this review must be maintained.

### 12.3 PTs

Proficiency tests for this method exist from AHIA in the form of PAT and AAR (Asbestos Analyst Registry) samples, and also from NY State ELAP.

### 12.4 Accuracy

There is no independent means of assessing the overall accuracy of this method, although it may be possible to do so utilizing large numbers of inter-laboratory analyses of a single sample. EMSL attempts this by reanalysis of reference slides on a daily basis. Regardless, it shall be noted analysis of lightly loaded samples <100 f/mm<sup>2</sup> is not as accurate as analysis of samples in the recommended loading range (100-1300 f/mm<sup>2</sup>), and are most likely an overestimate of the actual density.

### 12.5 Precision

Precision is determined per analyst over loading ranges 5-20, 21-50 and >51 fibers per 100 fields as the coefficient of variance (also called relative standard deviation). An overall laboratory precision is also calculated.

## 13.0 Quality Control

**13.1** All QC data must be maintained and available for easy reference and inspection.

**13.2** QC that is part of a new analyst's training cannot be used as QC for real time samples.

**13.3** Field blanks, when provided, are analyzed and sample results are blank corrected. Field blanks shall be submitted at a rate of 10%, or a minimum of 2 per sample set.

**13.4** Reference samples are analyzed daily per analyst for any day an analyst is analyzing samples.

**13.4.1** A minimum of two (2) reference samples per range (6 total) will be read monthly for NYS ELAP accredited labs.

**13.5** Intra-analyst QC is performed at a rate of 10% (excluding blanks).

**13.6** Inter-analyst QC is performed with this method through round robins, certain PT samples and training.

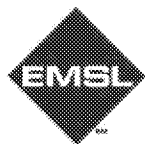
**13.7** Inter-laboratory QC in the form of round robins shall be conducted.

## 14.0 Data Assessment

**14.1 Acceptance Criteria for QC Measures:** These are addressed in EMSL's QMS Manual, Module A.

**14.1.1** Recounts are compared using the analyst's CV for the fiber range in question and formula 10.5 in the Calculations section.

**Note:** When recounts are performed during analyst training, recounts shall be compared using the laboratory's current overall CV for the fiber range in question, and formula 10.5 in the Calculations section.



ASB-SOP-300 PCM NIOSH 7400

Revision 17

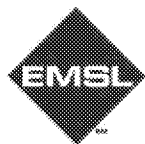
Revision Date: July 22, 2019

Effective Date: August 2, 2019

- 14.1.2** If a real time sample falls outside the acceptable limits:
- 14.1.2.1** It needs to be reconciled with the analyst and/or a 2<sup>nd</sup> analyst, when necessary.
  - 14.1.2.2** If a non-analytical cause was determined (ie. analysis of the wrong sample, incorrect data entry, etc.), the data is corrected and the sample results are re-evaluated by the PCM recount QC evaluation criteria. If sample passes this test, report the results as normal.
  - 14.1.2.3** If a non-analytical cause for the failure cannot be found, all samples in the sample set are to be reanalyzed and tested by using the PCM recount QC evaluation formula for Pass/Failure.
  - 14.1.2.4** For samples that passed this QC test, report the original results.
  - 14.1.2.5** For samples that fail this QC test, re-prepare and recount all samples that failed QC. Perform recounts on all re-prepped samples, and report all passing samples as usual.
  - 14.1.2.6** Any sample results failing this second QC test, if reported, must include a sample comment noting the QC failure.
- 14.1.3** Reference slide analysis acceptance is determined:
- 14.1.3.1** For past proficiency samples, by comparing the daily analysis to the published acceptance limits for the sample
  - 14.1.3.2** For past real time samples, by comparing the daily analysis to the calculated limits for the sample, after multiple analyses to determine mean, standard deviation, and limits
  - 14.1.3.3** Analysis of samples may not start until a satisfactory reference sample analysis is completed on a daily basis.
  - 14.1.3.4** Failed analyses are not removed from the data pool.
- 14.1.4** Field blanks containing more than seven (7) fibers per 100 graticule fields are noted with a report comment as being possibly contaminated, and are not used for blank correction.
- 14.2** **Corrections:** These policies are addressed fully in EMSL's QMS Manual, Module A.
- 14.2.1** All corrective actions shall look for the root cause of the error.
  - 14.2.2** All out-of-control or unacceptable data must be brought to the attention of the laboratory manager.
  - 14.2.3** The laboratory or QA manager is responsible for generating a corrective action, including an investigation of calibration procedures, a review of analytical techniques, and an investigation of training policies and compliance.
  - 14.2.4** Corrective actions will be reported to the QA department by means of the Quarterly Management Report, or sooner when appropriate.
- 14.3** **Contingencies for Handling Out-of-control or Unacceptable Data:**
- Any quality control requirements not met must have an explanation to their nonconformance.

## 15.0 Method Validation

- 15.1** EMSL Analytical Inc. uses analytical methods published or approved by regulatory authorities or consensus standard development agencies. Additional method validation is performed by use. Quality control data is evaluated on a regular basis to ensure effectiveness of the procedure.



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

## 16.0 Pollution Prevention / Waste Management

### 16.1 Pollution Prevention

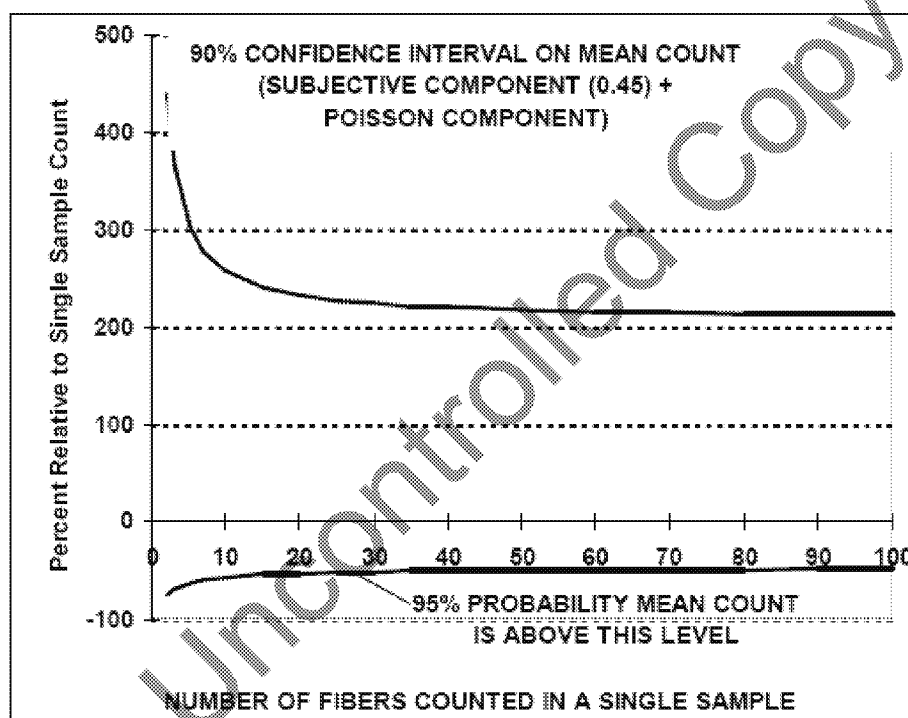
EMSL Analytical makes all efforts to reduce the volume and toxicity of the waste generated by the laboratory. An effort to manage procurement of hazardous materials has been implemented in order to avoid over ordering. Hazardous waste is classified for proper disposal.

### 16.2 Waste Management

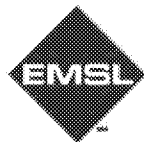
The waste generated during prep and analysis will be disposed of following safety procedures outlined in the chemical hygiene and safety plan.

## 17.0 Tables, Diagrams, Flowcharts, and Validation Data

### 17.1 Example: Confidence Limits for NIOSH 7400







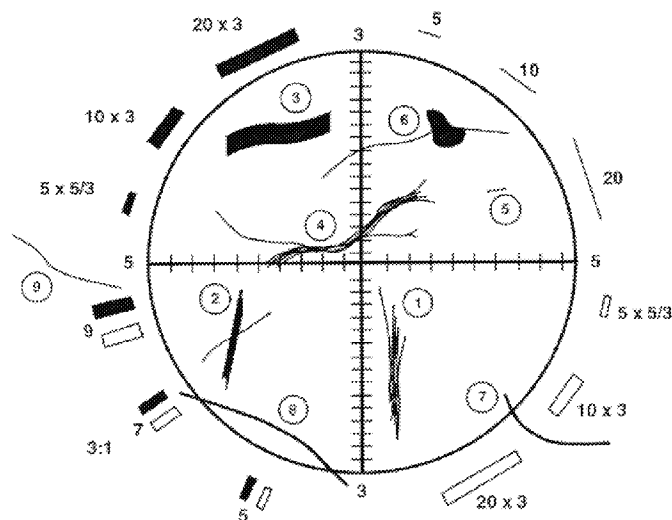
ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

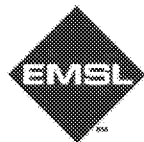
Effective Date: August 2, 2019

## 17.2 Example: Fibers and Counts



### FIBER COUNT

Object	Count	DISCUSSION
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fiber	If fibers meeting the length and aspect ratio criteria (length $\geq 5 \mu m$ and length-to-width ratio $\geq 3$ to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter ( $> 3 \mu m$ ), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is $< 5 \mu m$ long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	1/2 fiber	A fiber which crosses into the graticule area one time is counted as 1/2 fiber.
8	Do not count	Ignore fibers that cross the graticulate boundary more than once.
9	Do not count	Ignore fibers that lie outside the graticule boundary.



ASB-SOP-300 PCM NIOSH 7400  
Revision 17  
Revision Date: July 22, 2019  
Effective Date: August 2, 2019

## 18.0 References

- 18.1 NIOSH 7400 - Asbestos and Other Fibers by PCM Issue 3 – June 14, 2019.
- 18.2 Less is Better - Guide to Minimizing Waste in Laboratories prepared by the Task Force on Laboratory Environment, Health and Safety - American Chemical Society 2002.
- 18.3 EMSL Quality Management System Manual
- 18.4 EMSL Chemical Hygiene and Safety Plan SAF-SOP-2
- 18.5 ISO/IEC 17025:2017 General Requirements for the Competence of Testing and Calibration Laboratories.
- 18.6 American Hygiene Association: *Quality Assurance Manual for Industrial Hygiene Chemistry*. Fairfax, VA, American Industrial Hygiene Association.

## 19.0 Revision History

Revision	Rev. Date	Changes in this Revisions	Initials
17	7/22/2019	Revised per changes made to NIOSH 7400 Issue 3 – June 14, 2019 (see yellow highlights) Sample prep Section 9.3 amended extensively; adding optional clearing, mounting procedures Updated document names throughout; references in Section 18; QC form examples	MM
16	1/19/2017	Modified acceptance criteria in Section 8.3.5.1 for HSE/NPL Mark II Slide to allow 4th and 5th set of lines to be either completely or partially visible 11.18.1, 11.18.2: Added reporting of inter and intra-laboratory Sr values on reports Section 11.20.3: Clarified uncertainty calculations are derived from NIOSH 7400 Method	KN
15.2	11/04/2016	Section 9.5: Added note NYS ELAP doesn't recognize B Rules as part of their accreditation Added Section 9.3.8 to address one month hold time for preparations	KN

### Authorizing Signatures

Michelle McGowan  
Author/Revised by (print)

Author Signature

7/22/2019  
Date

Ed Cahill  
QA Reviewer (print)

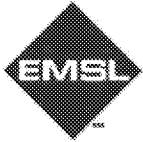
Reviewer Signature

7/23/2019  
Date

Kenneth Najuch  
Corporate Approval (print)

Corporate Approval Signature

7/23/2019  
Date



ASB-SOP-300 PCM NIOSH 7400

Revision 17

Revision Date: July 22, 2019

Effective Date: August 2, 2019

# Document Review and Acknowledgement Form

**Instructions: Ensure all personnel in laboratory affected by this document read and acknowledge receipt of document. Once completed, page shall be retained on file along with personnel training.**

### Laboratory Staff Acknowledgement

My signature below signifies that I have read, understood, and have had the opportunity to ask questions regarding the entire contents of this document. I agree to fully comply with, implement, and enforce all requirements, procedures, and protocols specified in this document and any supporting reference materials or methodologies.

## Print Name

**Signature**

Date \_\_\_\_\_

Uncontrolled Copy

**Acknowledged by:**

Lab Manager/QA Officer (Print Name)

Signature

Date \_\_\_\_\_



Calscience

## 2018 SOP Review

Group Leader Name (Print): Irina Semenova

Page: 1 of 2

Test Method or Subject	SOP Number	Effective Date	Last Review		Reviewer Chemist ID(s)	Review	
			Date	Revision		Date	Revision
EPA 3005A	M220	2015-03-23	2017-06-02	4.0	WLGG	08/29/18	
EPA 3020A	M221	2012-11-12	2017-06-02	3.2	WLGG	08/29/18	
EPA 3050B → D4	M222	2012-11-12	2017-06-02	1.2	WLGG JIAS 08/29/18	08/29/18	
EPA 3010A	M223	2015-03-26	2017-06-02	3.3	WLGG	08/29/18	
Reductive Precipitation	M225	2009-01-12	2017-06-02	1.0	UFLG	8/29/18	
EPA 1311 TCLP	M226	2016-05-04	2017-06-02	3.1	UJAS	08/27/18	
EPA 1312 SPLP	M227	2012-03-30	2017-06-02	1.3	UJAS	08/27/18	
CCR T22 WET (STLC)	M228	2012-12-10	2017-06-02	1.4	UJAS	08/27/18	
Homogenization/Compositing/Subsampling Solids	M230	2014-01-17	2017-06-02	2.2	UJAS	08/27/18	
EPA 7000 (FLAA)	M600	2005-11-25	2017-06-02	2.4	UFLG	8/29/18	
EPA 6010B → D4	M601	2016-05-02	2017-06-02	6.2			
Organic Lead (LUFT)	M602	2012-11-19	2017-06-02	2.2	F40Y	8/29/18	
EPA 1640	M609	2013-09-23	2017-06-02	1.1	UFLG	8/29/18	
EPA 6020 → D4	M610	2014-06-23	2017-06-02	4.3			
EPA 200.8 → D4	M611	2014-07-07	2017-06-02	1.2			
EPA 7471B	M618	2012-06-29	2017-06-02	0.0	DT-N23V	8/29/18	
EPA 7470A	M619	2015-04-06	2017-06-02	3.1	DT-N23V	8/29/18	
EPA 7471A	M620	2015-04-06	2017-06-02	3.6	DT-N23V	8/29/18	
EPA 245.1	M621	2007-09-26	2017-06-02	0.0	DT-N23V	8/29/18	
Lead in SPM	M623	2012-08-06	2017-06-02	1.0	UJAS	08/28/18	
EPA 200.7 → D4	M624	2013-06-17	2017-06-02	1.0			

2016-05-02 Revision

Eurofins Calscience, Inc.

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Page: 2 of 2

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STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 1 of 14

Title : 40 CFR, PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR

Document No. : SOP-M623  
Revision No. : 1.0  
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**Revision 1.0 changes encompass the entirety of the document.**

APPROVED FOR RELEASE BY:

  
MANAGEMENT

  
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QA DEPARTMENT

  
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Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 2 of 14

## 1. METHOD IDENTIFICATION

- 1.1. 40 CFR, PART 50, APPENDIX "G", Determination of Lead in Suspended Particulate Matter Collected from Ambient Air.

## 2. APPLICABLE MATRICES

- 2.1. This standard operating procedure is used to subsample and then prepare the subsample for the determination of lead, or other metals, in suspended particulate matter.
- 2.2. Glass fiber particulate filters, approximately 8" x 10", are the primary sample collection device.

## 3. DETECTION LIMITS

- 3.1. The estimated quantitation limits (EQLs) are defined by the analytical method: EPA Method 6010 or EPA Method 6020, as applicable.
  - 3.1.1. Results are reported on a whole filter basis.
  - 3.1.2. The analytical results are reported as ug/sample.
- 3.2. Refer to the appropriate analytical SOP for specific MDLs or RLs.

## 4. SCOPE AND APPLICATION

- 4.1. This method defines the subsampling procedure for samples that are to be analyzed for the mass of lead in the particulate matter collected on high-volume filters used in high-volume air samplers in ambient air testing.
  - 4.1.1. Whereas the 40CFR, Part 50, Appendix "G" method is written specifically for the determination of lead, and defines both a preparation and analytical procedure, this SOP covers only the subsampling procedure used prior to digestion and analysis.
  - 4.1.2. The analytical method chosen may also be used to identify and quantify other elements that may be present in the particulate matter as noted in the specific method.
- 4.2. This method is restricted to use by or under the supervision of analysts experienced in the use of related subsampling, digestion and analytical equipment.

## 5. METHOD SUMMARY

- 5.1. Particulate filters are subsampled using a Teflon template and ceramic cutting device prior to digestion using Calscience SOP M222: EPA Method 3050: Acid Digestion of Sediments, Sludges and soils.

STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 3 of 14

- 5.2. Following digestion, the sample is analyzed by ICP or ICPMS using EPA Method 6010 or EPA Method 6020, respectively.

## 6. DEFINITIONS

- 6.1. Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.
- 6.2. Batch, Preparation: Environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria.
- 6.3. Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.
- 6.4. Holding Times: The maximum times that samples may be held prior to analysis and still be considered valid or not compromised.
- 6.5. Laboratory Control Sample: A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes. It is used to establish intra-laboratory or analyst-specific precision and bias or to assess the performance of all or a portion of the measurement system.
- 6.6. Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
- 6.7. Limit of Detection (LOD): A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility.
- 6.8. Limit of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
- 6.9. Matrix Spike: A sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
- 6.10. Matrix Spike Duplicate: A second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
- 6.11. Method Blank: A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
- 6.12. Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.



STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 4 of 14

- 6.13. Quality Assurance: An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.
- 6.14. Quality Control: The overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.
- 6.15. Raw Data: Any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

## 7. INTERFERENCES

- 7.1. Two types of interferences are possible: Chemical and light scattering. Chemical interferences are rare, but if suspected, the analytical portion associated with the samples should be carried out with and without the method of standard additions. If requested or directed by the client, the method of standard additions must be used.
- 7.2. As the analysis portion follows EPA Method 6010 or EPA Method 6020, interferences specific to those methods may be seen. Follow the specific determinative SOP for information related to potential analytical interferences.
- 7.3. Light scattering is produced by high concentrations of dissolved solids in the sample, and can produce a significant interference, especially at low lead concentrations.

## 8. SAFETY

- 8.1. Many metal salts are extremely toxic if inhaled or swallowed. Extreme care must be taken to ensure that samples are handled properly and that all exhaust gases are properly vented. Wash hands thoroughly after handling.
- 8.2. Sharp cutting devices are used in this procedure. Use extreme care when cutting the filters.
- 8.3. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Calscience's Health, Safety, and Respiratory Protection Manual. At a minimum, safety glasses, gloves and a laboratory coat are required to be worn while working in the lab.
- 8.4. Dilute acids will be used in the equipment cleaning steps and the digestion step following this subsampling procedure. Use extreme caution and wear appropriate

STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 5 of 14

personal protective equipment at all times when working with and around acids. Precautions must be taken to avoid inhalation, ingestion, or skin contact.

- 8.5. Material Safety Data Sheets (MSDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the MSDS for all chemicals to be used prior to handling.

## 9. EQUIPMENT AND SUPPLIES

- 9.1. Calibrated balance, capable of weighing to 0.1g.
- 9.2. Digestion tubes: 4oz (120mL), 90mm x 43mm ID, graduated, snap closure, with hinged lids, polypropylene, disposable, Capitol Vial P/N 04HPLS or equivalent.
- 9.3. Cutting device tool, Fine Science Tools # 10025-50 or equivalent
- 9.4. Ceramic Blades, Fine Science Tools # 10025-45 or equivalent
- 9.5. ¾" x 8" Teflon template: multiple templates of the same size, individually numbered
- 9.6. Nitrile or powder-free latex gloves
- 9.7. Forceps, plastic or teflon coated
- 9.8. Clean bench pad (optional – Teflon cutting board at least 12" x 16")
- 9.9. Clean paper towels

## 10. REAGENTS AND STANDARDS

- 10.1. Reagents
  - 10.1.1. Reagent water; distilled or deionized
  - 10.1.2. Nitric acid (HNO<sub>3</sub>), 0.5% (v/v). Used for washing subsampling equipment between uses.
    - 10.1.2.1. 0.5% HNO<sub>3</sub> (v/v): Prepare by adding 0.5 parts 70-71 percent concentrated HNO<sub>3</sub>, reagent grade, to 99.5 parts DI water.

## 11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

- 11.1. Samples should be collected by field personnel experienced in handling particulate filters.
- 11.2. The following sample collection procedures are from 40CFR, Part 50, Appendix "B", Section 8. These are suggested procedures only, as actual sample collection procedures may be otherwise defined by method or regulation.
  - 11.2.1. Stop the sampler and carefully remove the filter, following the sampler manufacturer's instructions. Touch only the outer edges of the filter.

STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 6 of 14

- 11.2.2. Fold the filter in half lengthwise so that only surfaces with collected particulate matter are in contact and place it in the filter holder (glassine envelope or manila folder).
- 11.3. Wear clean gloves whenever handling a new filter to limit the potential for cross contamination.
- 11.4. Upon receipt, the samples are stored in a 4°C cooler.
- 11.5. Samples must be analyzed within 180 days of sample collection.

## 12. QUALITY CONTROL

- 12.1. Multiple subsamples from the same filter shall be cut using the same Teflon template and shall be cut at the same time by the same technician.
- 12.2. Field Blank: A blank filter, if provided by the client, shall be subsampled at the same time, in the same manner by the same technician as the associated samples.
- 12.3. Sample Based QC
  - 12.3.1. As used within this SOP, a batch shall mean 20 field samples.
  - 12.3.2. Quality Control must be performed for every batch of samples prior to the analysis of the samples. Quality control consists of, at a minimum, a Method Blank (MB) and a laboratory control sample (LCS) and its duplicate (LCSD). Additionally, matrix based QC (MS/MSD) may also be prepared at the same time as the field samples.
    - 12.3.2.1. The MB consists of a clean filter strip. If a blank filter strip is not available, follow the QC procedures outlined in Calscience SOP M222 for the preparation of a Method Blank.
    - 12.3.2.2. The LCS and LCSD will consist of a lead standard spiked onto two additional clean filter strips. If blank filter strips are not available, follow the QC procedures outlined in Calscience SOP M222 for the preparation of an LCS/LCSD.
    - 12.3.2.3. The MS and MSD will consist of a lead standard spiked onto two additional field sample strips and will be processed in the same manner as the field samples.
- 12.4. Refer to the appropriate digestive and/or determinative method for related analytical quality control information.
- 12.5. Additional information regarding internal QC checks is provided in SOP-T020.

## 13. CALIBRATION AND STANDARDIZATION

- 13.1. Refer to the appropriate determinative method for related analytical quality control information.
- 13.2. Analytical Balance

STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/08/12  
Page 7 of 14

- 13.2.1. Calibrate the balance at 0.1g, 1g, and 100g using Class 2 weights.
- 13.2.2. Calibration shall be within  $\pm 10\%$  at 0.1g ( $\pm 0.01g$ ), or within  $\pm 2\%$  at 1g ( $\pm 0.02g$ ) and at 100g ( $\pm 2g$ ). If the values are not within these limits, recalibrate the balance.

#### 14. PROCEDURE

- 14.1. Retrieve samples from sample receiving. Samples will be in approximately 10" x 12" manila envelopes.
- 14.2. Place a layer of clean bench padding down on top of the working surface of the bench. As the ceramic blade is very sharp, this padding will help prevent the blade from penetrating all the way to the bench top. If available, a Teflon cutting board may be used in place of the padding.
- 14.3. Place two to three layers of paper towels on the bench padding. (The filters will be placed on top of the towels for cutting.)
  - 14.3.1. The top paper towel sheet should be changed out with a clean sheet, or another sheet added on top of the others, between cutting each filter to limit cross contamination.
- 14.4. Carefully open up the envelope and retrieve the filter. The client should have folded the filter in half lengthwise prior to placing inside the envelope.
  - 14.4.1. It is suggested that clean plastic forceps be used when handling the filter and that the filters are only handled by the edges.
  - 14.4.2. If the filters will be handled by gloved hands, be sure to change gloves prior to handling the next filter, or if the gloves become contaminated at any time during the process. Again, handle the filter only by the edges.
  - 14.4.3. Place the folded filter on top of the clean paper towels. Lay the filter lengthwise so that the folded part is at the top when laid out on the towels.
    - 14.4.3.1. Alternately a cutting apparatus may be constructed to facilitate high volume sample processing, reference Appendix A.
- 14.5. Retrieve a knife, with clean ceramic blade, and a clean numbered Teflon template. Note the template number in the comments section of the solid sample preparation logbook.
- 14.6. Because the particulate matter is contained on all but approximately a  $\frac{1}{2}$ " border around the filter, measure in from the left side edge of the filter at least one inch before placing the template on the filter. The left edge of the filter, or right edge if multiple subsamples are required, will not be used in the digestion and analytical processes.
- 14.7. Lay the Teflon template in a vertical direction across the folded filter. In order to cut the straightest lines on the filter, line up the top of the template with the fold. The bottom of the template will hang off the bottom of the filter. Cutting the filter in this manner will provide the most representative subsample.

STANDARD OPERATING PROCEDURE

Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 8 of 14

- 14.8. Pressing firmly on the template so it does not move, cut along both sides of the template using a single motion from top to bottom. The filters should cut easily so use only enough pressure to make a clean cut.
  - 14.8.1. **NOTE:** The ceramic blades are extremely sharp, be careful not to cut the template. Using a an acute angle (i.e., toward the table top) on the cutting device will help prevent this.
  - 14.8.2. If only one strip is needed, use forceps to remove the pieces of filter that will not be used and return them to the manila envelope. Handle the filter pieces only by the edges and be sure to return to the correct envelope. Leave the folded strip on the paper towel for further processing as per section 14.13.
- 14.9. If more than one subsample is needed, use plastic forceps to grasp the folded strip by the bottom edge and set aside on a clean paper towel to await further processing.
  - 14.9.1. At this point a clean ceramic blade should be used for each additional subsample. At a minimum, the blade should be wiped off with a clean chem wipe between each cut.
  - 14.9.2. The blade can be rinsed with a dilute nitric acid solution, followed by DI water and then wiped dry with a chem wipe. Be sure the blade is completely dry before making additional cuts if this procedure is used. It is suggested that more than one blade/knife be available if multiple samples are being subsampled to expedite the process.
- 14.10. To cut the filter for additional MS/MSD or sample duplicate strips, move the template over, lining up the top with the fold and left edge of the template with the previous cut. Press firmly on the template and make another cut along the right side of the template. Continue this process until the number of subsamples needed have been cut. Set aside each strip onto the clean paper towel as done with the first subsample. Leave the strips folded in half.
  - 14.10.1. Do not place the strips on top of one another. Once cut, handle each strip individually to limit cross contamination.
  - 14.10.2. Remove the template from the filter and set aside for cleaning. A clean template must be used for each additional sample. The template should be rinsed with a dilute nitric acid solution, followed by DI water and then wiped dry with a chem wipe. Be sure the template is completely dry before placing on the next filter sample. It is recommended that more than one template be available if multiple samples are being subsampled to expedite the process.
- 14.11. Once all cuts have been made, return the unused filter pieces to the manila envelope. Handle all pieces by the edges.
- 14.12. Take a folded strip and return to the cutting surface, place on top of a clean paper towel. Using a clean blade, cut the strip into smaller pieces, approximately ½ inch sections, to aid in the digestion process.
- 14.13. Label a digestion tube with the Calscience sample ID.

STANDARD OPERATING PROCEDURE

Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 9 of 14

- 14.14. Put all the pieces of the subsample into the digestion tube and record the final area of the whole sample to the preparation logbook based on the Teflon® template size of 3/8" x 8". Use clean plastic forceps to handle the pieces.

- 14.14.1. As the filter is folded in half, and the back of the filter (the portion without particulate matter) is exposed, use the forceps to pick up the cut pieces without touching the particulate matter. In essence you are picking up two pieces at a time, where the particulate matter is sandwiched in between the two pieces.

- 14.15. Cap the digestion tube and set aside.

- 14.16. Digestion will follow the procedures outlined in Calscience SOP M222, EPA 3050: Acid digestion of Sediments, Sludges and soils.

- 14.17. Record all aspects of the subsampling procedure in the sample preparation logbook.

- 14.18. Thoroughly clean the ceramic blades, templates, forceps and Teflon cutting board (if used) with a dilute nitric acid solution followed by a DI rinse and allow to dry. Once dry, place these items into a plastic bag or cardboard box for storage.

## 15. CALCULATIONS

- 15.1. Refer to the specific determinative method SOP for information related to calculations.

- 15.2. Sample results are reported based upon the whole filter.

- 15.2.1. Analytical results from the analytical method are multiplied by a conversion factor of 100 (EPA 6010) or 2000 (EPA 6020) and are then multiplied by 12.

- 15.2.2. Sample results are reported in ug/sample units.

## 16. METHOD PERFORMANCE

- 16.1. An initial demonstration of analytical capability shall be performed for the digestion and analytical methods per the procedures outlined in the specific preparation and determinative SOPs.

- 16.2. Calibration protocols specified in Section 13, "Calibration and Standardization," shall be followed.

## 17. POLLUTION PREVENTION

- 17.1. The toxicity, carcinogenicity and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.

- 17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Calscience's Health, Safety, and Respiratory Protection Manual. In

STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 10 of 14

general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.

- 17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:

17.3.1. A NIOSH approved air purifying respirator with cartridges appropriate for the chemical handled.

17.3.2. Extended length protective gloves.

17.3.3. Face shield.

17.3.4. Full-length laboratory apron.

- 17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.

- 17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.

## 18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

- 18.1. Refer to the specific determinative method SOP for information related to data assessment and acceptance criteria as well as reporting units and significant figures.

- 18.2. Additional information regarding internal QC checks is provided in SOP-T020.

## 19. CORRECTIVE ACTIONS

- 19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.

- 19.2. The Operations Manager, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.

- 19.3. Corrective actions are generally of two types, immediate and long-term actions.

STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 11 of 14

- 19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.
- 19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:
  - 19.3.2.1. Remedial training of staff in technical skills, technique or implementation of operating procedures.
  - 19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.
  - 19.3.2.3. Revision of standard operating procedures.
  - 19.3.2.4. Replacing personnel, as necessary.
- 19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:
  - 19.4.1. Define the problem.
  - 19.4.2. Assign responsibility for investigating the problem.
  - 19.4.3. Investigate and determine the cause of the problem.
  - 19.4.4. Assign and accept responsibility for implementing the corrective action.
  - 19.4.5. Determine effectiveness of the corrective action and implement correction.
  - 19.4.6. Verify that the corrective action has eliminated the problem.
- 19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

## 20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

- 20.1. Refer to the specific determinative method SOP for information related to out-of-control or unacceptable data.

## 21. WASTE MANAGEMENT

- 21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated,



**STANDARD OPERATING PROCEDURE**

Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623

Revision No.: 1.0

Effective Date: 08/06/12

Page 12 of 14

and either returned to the client (preferable) or placed into the proper laboratory waste stream.

- 21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.
- 21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.
- 21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.
- 21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.
- 21.6. In order to maintain accountability for all samples received by Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.
- 21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

## 22. REFERENCES

- 22.1. Title 40: Protection of the Environment, Appendix G to Part 50 – Reference Method for the Determination of Lead in Suspended Particulate Matter Collected From Ambient Air.
- 22.2. Title 40: Protection of the Environment, Appendix B to Part 50 – Reference Method for the Determination of Suspended Particulate Matter in the Atmosphere (High-Volume Method).
- 22.3. Calscience SOP M222, EPA 3050: Acid Digestion of Sediments, Sludges and soils.

## 23. APPENDICIES

- 23.1. Appendix A: Dedicated Cutting Apparatus

## 24. MODIFICATIONS

Calscience SOP M623 Section	Reference Document Appendix G to Part 50 Section	Summary of Modification
14.0, 15.0	6.2	Substitute EPA 3050, EPA 6010 and EPA 6020 digestive and analytical methods.

STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 13 of 14

## 25. REVISION HISTORY

Revision	Description	Author	Effective Date
SOP 143	SOP Created based on 40 CFR, Part 50, Appendix G	B. Christensen	03/02/1995
1.0	Revised to NELAP format.	L. Scharpenberg	08/06/2012

STANDARD OPERATING PROCEDURE  
Title: 40 CFR PART 50, APPENDIX G, (M) DETERMINATION OF LEAD IN  
SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR  
Calscience Environmental Laboratories, Inc.

Document No.: SOP-M623  
Revision No.: 1.0  
Effective Date: 08/06/12  
Page 14 of 14

## APPENDIX A DEDICATED CUTTING APPARATUS

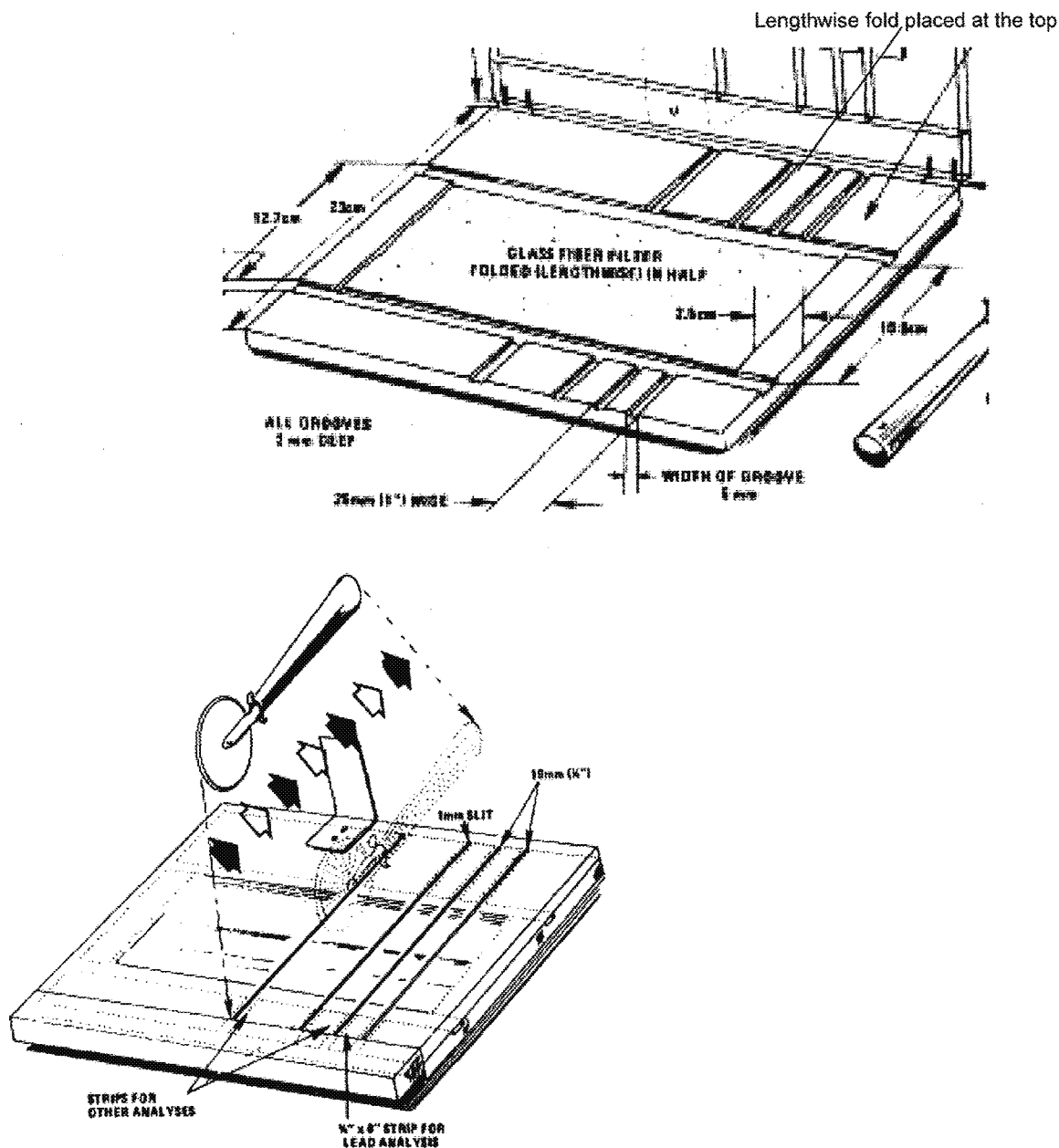


Figure 2

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 1 of 70

Title : EPA METHOD 6010B, INDUCTIVELY COUPLED PLASMA –  
ATOMIC EMISSION SPECTROMETRY (ICP-AES)

Document No. : SOP-M601  
Revision No. : 6.2  
Supersedes : 6.1

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Revision 6.2 changes are noted in bold italicized typeface and preceded by a "►" marker.

APPROVED FOR RELEASE BY:

  
MANAGEMENT

5/2/2016  
DATE

  
QA DEPARTMENT

04-29-16  
DATE

Reviewer Signature	Review Date	Comments	QA Signature

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 2 of 70

## 1. METHOD IDENTIFICATION

- 1.1. EPA Method 6010B, Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES).

## 2. APPLICABLE MATRICES

- 2.1. This method is applicable to groundwater samples, aqueous samples, mobility-procedure extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes.

## 3. DETECTION / QUANTITATION LIMITS

- 3.1. The RLs will be proportionally higher for samples which require dilution or reduced sample size.
- 3.2. Refer to the current revision of SOP-T006, Determination of Detection Limits, for procedure on establishing detection and reporting limits.
  - 3.2.1. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions.
  - 3.2.2. The instrument detection limit data may be used to estimate instrument and method performance for other sample matrices.

## 4. SCOPE AND APPLICATION

- 4.1. EPA Method 6010B is used to determine trace elements, including metals, in solution. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludges, sediments, and other solid wastes, require acid digestion prior to analysis.
  - 4.1.1. Groundwater samples that have been prefiltered and acidified will not need acid digestion.
  - 4.1.2. Samples which are not digested must either use an internal standard or be matrix matched with the standards. If either option is used, instrument software should be programmed to correct for intensity differences of the internal standard between samples and standards.
- 4.2. The method is applicable to the elements listed in Appendix A. Appendix A also lists the recommended analytical wavelengths and estimated instrument detection limits for the elements in clean aqueous matrices with insignificant background interferences. Elements and matrices other than those listed in Appendix A may be analyzed by this method if performance at the concentrations of interest (see Section 12.) is demonstrated.
- 4.3. This method is restricted to use by or under the supervision of analysts experienced in the use of inductively coupled plasma emission spectrometer, skilled in the

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 3 of 70

interpretation of atomic emission spectra, and knowledgeable in the correction of spectral, chemical, and physical interferences described in this method.

## 5. METHOD SUMMARY

- 5.1. EPA Method 6010B describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices.
- 5.2. Background correction is required for trace element determination. Background emission must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used should be as free as possible from spectral interference and should reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences identified in Section 7. should also be recognized and appropriate corrections made. Alternatively, multivariate calibration methods may be utilized. In this case, point selections for background correction are superfluous since whole spectral regions are processed.
- 5.3. Prior to analysis, samples must be solubilized or digested using the appropriate sample preparation methods. Acceptable preparatory methods include, but are not limited to, the following:

<u>Type of Sample Preparation</u>	<u>EPA Method No.</u>	<u>SOP No.</u>
Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by FLAA/ICP	3005	SOP-M220
Acid Digestion of Aqueous Samples/Extracts for Total Metals for Analysis by FLAA/ICP	3010	SOP-M223
Acid Digestion of Sediments, Sludges, and Soils	3050	SOP-M222
Toxicity Characteristic Leaching Procedure (TCLP)	1311	SOP-M226
Synthetic Precipitation Leaching Procedure (SPLP)	1312	SOP-M227
Waste Extraction Test Procedure (STLC/TTLC)	CCR T22.11.5.A-II	SOP-M228

- 5.4. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

## 6. ►DEFINITIONS

### 6.1. Terms Specific to ICP-AES Analysis

- 6.1.1. Dissolved Metals: The concentration of metals determined in an aqueous sample after the sample is filtered through a 0.45-µm filter.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 4 of 70

- 6.1.2. Instrument Detection Limit (IDL): A tool for evaluating the instrument noise level and response changes over time for analytes of interest. IDLs can be estimated by calculating the average of the standard deviations of three analytical runs performed on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement should be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples).
- 6.1.3. Interference Check Sample (ICS): A solution containing both interfering and analyte elements of known concentration that can be used to verify background and inter-element correction factors.
- 6.1.4. Linear Dynamic Range: The concentration range above the highest calibration point over which the functional relationship between analyte signal and analyte concentration remains linear based on a one-point calibration. A sample result that falls within the linear dynamic range is considered valid and may be reported, thus avoiding the need to dilute and re-analyze the sample.
- 6.1.5. Method of Standard Addition (MSA): An alternative calibration procedure employed when the signal response of the analyte of interest is different in a particular matrix than when it is in reagent water. The procedure is generally reserved for analyzing complex matrices. The standard addition technique involves the addition of known amounts of the target analyte to each of a series of replicate sample aliquots. The final concentrations of the sample replicates should span the calibration range of the method. The analytical response versus the standard addition concentration for each of the replicates is plotted. After performing a linear regression, the curve is extrapolated to the x-axis. The analyte concentration in the original unspiked sample is equal to the inverse of the x-intercept.
- 6.1.6. Optimum Concentration Range: A concentration range, below which scale expansion must be used, and above which curve correction should be considered. This range will vary with the sensitivity of the instrument and the operating conditions employed.
- 6.1.7. Post Digestion (Matrix) Spike: A sample which has been extracted in the same manner as the other samples, but to which a known amount of target analytes has been added to the sample extractant. Post digestion spikes are used to evaluate the accuracy of the method without the losses incurred through the extraction process.
- 6.1.8. Sensitivity: The average of the standard deviations of three runs of a reagent blank solution on three non-consecutive days with seven consecutive measurements per day.
- 6.1.9. Suspended Metals: The concentration of metals determined in the portion of an aqueous sample that is retained by a 0.45- $\mu$ m filter.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 5 of 70

- 6.1.10. Total Recoverable Metals (Total Acid Soluble Metals): The concentration of metals determined in an unfiltered sample following digestion using hot mineral acid. "Total recoverable metals" is referred to herein as "total metals."
- 6.2. Refer to the current revision of the Eurofins Calscience Quality Systems Manual for additional definitions *and glossaries*.

## 7. INTERFERENCES

- 7.1. Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.
- 7.1.1. Compensation for background emission and stray light can usually be conducted by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.
- 7.1.2. To determine the appropriate location for off-line background correction, the analyst must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single-element solutions are sufficient. However, for analytes such as iron that may be found in the sample at high concentration, a more appropriate test would be to use a concentration near the upper limit of the analytical range.



STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 6 of 70

- 7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require that the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive or positively biased determinations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Analysts may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off-line or on-line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelengths are given in Appendix B. For multivariate calibration methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.
- 7.1.4. When using interelement correction equations, the interference may be expressed as analyte concentration equivalents (i.e., false positive analyte concentrations) arising from 100 mg/L of the interference element. For example, if As is to be determined at 193.696 nm in a sample containing approximately 10 mg/L of Al. According to Appendix B, 100 mg/L of Al will yield a false positive signal for an As level equivalent to approximately 0.01085 mg/L. Therefore, the presence of 10 mg/L of Al will result in a false positive signal for As equivalent to approximately 0.001085 mg/L. The analyst is cautioned that other instruments may exhibit somewhat different levels of interference than those shown in Appendix B. The interference effects must be evaluated for each individual instrument, since the intensities will vary.
- 7.1.5. Interelement corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Interelement corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Interelement corrections that constitute a major portion of an emission signal may not yield accurate data. Analysts should continuously note that some samples may contain uncommon elements that could contribute spectral interferences.
- 7.1.6. The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 7 of 70

interferences (see Appendix B) as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst shall utilize a computer routine for automatic correction on all analyses.

- 7.1.7. Analysts using sequential instruments must verify the absence of spectral interference by scanning over a range of 0.5 nm centered on the wavelength of interest for several samples. The range for lead, for example, would be from 220.6 to 220.1 nm. This procedure must be repeated whenever a new matrix is to be analyzed and when a new calibration curve using different instrumental conditions is to be prepared. Samples that show an elevated background emission across the range may be background corrected by applying a correction factor equal to the emission adjacent to the line or at two points on either side of the line and interpolating between them. An alternate wavelength that does not exhibit a background shift or spectral overlap may also be used.
- 7.1.8. If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and dividing by 10. If after the subtraction of the calibration blank, the apparent analyte concentration falls outside of this range, in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.
- 7.1.9. When interelement corrections are applied, their accuracy should be verified daily, by analyzing spectral interference check solutions. The correction factors or multivariate correction matrices tested on a daily basis must be within the 20% criteria for 5 consecutive days. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation change occurs, such as one in the torch, nebulizer, injector, or plasma conditions. Standard solutions should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.
- 7.1.10. When interelement corrections are not used, verification of absence of interferences is required.
  - 7.1.10.1. One method is to use a computer software routine for comparing the determinative data to established limits for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration (i.e., greater than the analyte instrument detection limit), or a false negative analyte concentration, (i.e.,

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 8 of 70

less than the lower control limit of the calibration blank defined for a 99% confidence interval).

- 7.1.10.2. Another method is to analyze an interference check solution which contains similar concentrations of the major components of the samples ( $> 10$  mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is  $\geq 20\%$  of the analyte concentration, the analyte must be determined (1) using analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.
- 7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump, by using an internal standard, or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization by using a tip washer, by using a high solids nebulizer, or by diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance. This may be accomplished with the use of mass flow controllers. The dilution test (see Section 12.13.) will help determine if a physical interference is present.
- 7.3. Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.
- 7.3.1. The MSA should be used if an interference is suspected or a new matrix is encountered. When the MSA is used, standards are added at one or more levels to portions of a prepared sample. This technique compensates for enhancement or depression of an analyte signal by a matrix. It will not correct for additive interferences, such as contamination, interelement interferences, or baseline shifts. This technique is valid in the linear range when the interference effect is constant over the range, the added analyte responds the same as the endogenous analyte, and the signal is corrected for additive interferences.
- 7.3.2. An alternative to using the MSA is to use the internal standard technique. Add one or more elements that are both not found in the samples and verified to not cause an interelement spectral interference to the samples, standards, and blanks. Yttrium or scandium is often used. The concentration should be sufficient for optimum precision, but not so high as

STANDARD OPERATING PROCEDURE

Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)

Eurofins Calscience, Inc.

Document No.: SOP-M601

Revision No.: 6.2

Effective Date: 2016-05-02

Page 9 of 70

to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation. This technique is very useful in overcoming matrix interferences, especially in high solids matrices.

- 7.4. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the buildup of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized within an analytical run and suitable rinse times should be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample should be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to equal to or less than the method detection limit should be noted. Until the required rinse time is established, it is suggested that the rinse period be at least 60 seconds between samples and standards. If a memory interference is suspected, the sample must be re-analyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon the project specific data quality objectives (DQOs).
- 7.5. Analysts are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations should be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.
- 7.6. The dashes in Appendix B indicate that no measurable interferences were observed even at higher interferant concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.
- 7.7. Clean chemistry methods and procedures are necessary in reducing the magnitude and variability of the calibration blank.

## 8. SAFETY

- 8.1. Concentrated nitric and hydrochloric acids are moderately toxic and extremely irritating to skin and mucus membranes. Hence, precautions must be taken to avoid inhalation, ingestion, or skin contact.
- 8.2. Many metal salts are extremely toxic if inhaled or swallowed. Extreme care must be taken to ensure that samples and standards are handled properly and that all exhaust gases are properly vented. Wash hands thoroughly after handling.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 10 of 70

- 8.3. All sample and standard preparation activities should be performed in an operational fume hood appropriate for use with acids.
  - 8.3.1. The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides.
  - 8.3.2. All operational fume hoods are to remain energized continuously in order to minimize acidic atmospheric or toxic gas buildup.
- 8.4. Exposure to hazardous chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current version of Eurofins Calscience's Health, Safety, and Respiratory Protection Manual. In general, safety glasses and laboratory coats are required to be worn in all designated laboratory areas. Protective gloves shall be worn when handling chemicals.
- 8.5. ► Safety Data Sheets (SDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the SDS for all chemicals to be used prior to handling.

## 9. EQUIPMENT AND SUPPLIES

- 9.1. Inductively Coupled Argon Plasma Emission Spectrometer, PerkinElmer Optical Emission Spectrometer Optima 5300 DV, PerkinElmer Optical Emission Spectrometer Optima 7300 DV, or equivalent configured with the following components:
  - 9.1.1. Computer-controlled emission spectrometer with background correction.
  - 9.1.2. Radio-frequency (RF) generator compliant with FCC regulations.
  - 9.1.3. Mass-flow controller for argon nebulizer gas supply.
  - 9.1.4. Peristaltic pump.
  - 9.1.5. Autosampler, Perkin-Elmer AS 93plus Autosampler, PerkinElmer ESI SC-4 Autosampler, or equivalent.
- 9.2. Instrument Software
  - 9.2.1. Require a PC based data system or equivalent.
  - 9.2.2. ► PerkinElmer WinLab32 for ICP Version **5.3.0.0656**, **PerkinElmer Syngistix for ICP Version 1.0.1.1275**, or equivalent.
- 9.3. Instrument Maintenance and Troubleshooting
  - 9.3.1. Refer to the current revision of SOP-T066 and instrument hardware and software manuals for instrument maintenance and troubleshooting.
  - 9.3.2. Additional information can be found in the user manual or operating guide for the specific instrument.
- 9.4. ICP Torch and Nebulizer Gas: Argon, Ar, 99.998%, cryogenic liquid, Praxair Argon Cryogenic Liquid or equivalent.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 11 of 70

- 9.5. Purge Gas: Nitrogen, N<sub>2</sub>, 99.998%, cryogenic liquid, Praxair Nitrogen Cryogenic Liquid or equivalent.
- 9.6. Volumetric flasks, 100 mL, 500 mL, 1000 mL, or other capacity, glass, Class A.
- 9.7. Bottles, various sizes, PTFE fluoropolymers, polyethylene, or polypropylene, with screw caps.
  - 9.7.1. Acid clean previously unused bottles with 1% HNO<sub>3</sub> solution prior to use.
- 9.8. Autosampler vessels, 16-mm OD (15 mL capacity), translucent polypropylene, disposal.
- 9.9. Autosampler vessels, 30-mm OD (50 mL capacity), with screw caps, translucent polypropylene, disposal.
- 9.10. Pipettors, 10–100 µL, 100–1000 µL, 0.5–5.0 mL, and 1–10 mL, calibrated, adjustable volume, with disposable tip.
- 9.11. Refer to the specific SOPs of the preparatory methods for additional equipment and supplies.

## 10. REAGENTS AND STANDARDS

### 10.1. Reagents

- 10.1.1. Reagent water, interferant free.
- 10.1.2. Chips, Teflon.
- 10.1.3. Beads, glass.
- 10.1.4. Hydrochloric acid, HCl, 36.5–38.0% (v/v), concentrated, colorless to pale yellow liquid, trace metals grade for equivalent.
- 10.1.5. Hydrochloric acid, HCl, 1:1 (v/v).
  - 10.1.5.1. Prepare the 1:1 HCl solution by slowly adding 500 mL of concentrated HCl to 400 mL of reagent water and diluting to 1 L with additional reagent water.
- 10.1.6. Nitric acid, HNO<sub>3</sub>, 68.0–70.0% (v/v), concentrated, clear to yellow liquid, trace metals grade for equivalent.
- 10.1.7. Nitric acid, HNO<sub>3</sub>, 1:1 (v/v).
  - 10.1.7.1. Prepare the 1:1 HNO<sub>3</sub> solution by slowly adding 500 mL of concentrated HNO<sub>3</sub> to 400 mL of reagent water and diluting to 1 L with additional reagent water.
- 10.1.8. Rinse blank, HCl-HNO<sub>3</sub>-H<sub>2</sub>O, 1:1:8 (v/v/v).
  - 10.1.8.1. Prepare the rinse blank by slowly adding 1 part of concentrated HCl and 1 part of concentrated HNO<sub>3</sub> to 8 parts of reagent water.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 12 of 70

- 10.1.8.2. The rinse blank consists of 10% (v/v) HCl and 10% (v/v) HNO<sub>3</sub> in reagent water.
- 10.1.8.3. The rinse blank is used to flush the system between standards and samples to minimize memory interferences (see Section 7.4.).
- 10.1.9. All reagents must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.
- 10.2. ►Standards
  - 10.2.1. Stock Standard Solutions
    - 10.2.1.1. Pre-certified stock standard solutions (ultra-high purity grade or equivalent), each in sealed polyethylene bottles, containing various concentrations of target analytes are used to prepare calibration and check standards.
    - 10.2.1.2. Prior to preparing the calibration or check standards, analyze each stock standard solution separately to determine possible spectral interference or the presence of impurities.
  - 10.2.2. Initial Calibration Standard Solutions
    - 10.2.2.1. Dilute the appropriate volumes of the stock standards, concentrated HCl, and concentrated HNO<sub>3</sub> to the specified volumes with reagent water for initial calibration.
      - 10.2.2.1.1. If the addition of silver to the recommended acid combination initially results in a precipitate, then add the appropriate volume of reagent water and warm the flask until the solution clears. Cool and dilute to the appropriate final volume with reagent water. Higher concentrations of silver require additional HCl. Determine the stability the silver in the solution if necessary.
    - 10.2.2.2. Use the analyte and acid concentrations outlined in Appendix C as guidance to prepare the calibration standards **if the commercially prepared custom reference standard solutions are unavailable.**
  - 10.2.3. Blanks
    - 10.2.3.1. Calibration Blank (CB)
      - 10.2.3.1.1. Prepare the CBs by acidifying reagent water to the same concentrations of the acids found in the sample digestates prepared via EPA Method 3010.
      - 10.2.3.1.2. The CB consists of 5% (v/v) HCl and 6% (v/v) HNO<sub>3</sub> in reagent water.
      - 10.2.3.1.3. The CB is used to establish the zero point of the calibration curve.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 13 of 70

- 10.2.3.1.4. The CB is also used either as initial calibration blank (ICB) or as continuing calibration blank (CCB) to monitor contamination.
- 10.2.3.2. Method Blank (MB)
  - 10.2.3.2.1. Prepare the MBs using the appropriate sample preparation methods (see Section 5.3.).
  - 10.2.3.2.2. The MB is used to identify possible contamination resulting from either the acids or the equipment used during sample processing including filtration.
- 10.2.3.3. Both CB and MB are required for the analyses of samples prepared by any method other than EPA Method 3040.
- 10.2.4. Initial Calibration Verification (ICV) Solutions
  - 10.2.4.1. Dilute the appropriate volumes of the stock standards, concentrated HCl, and concentrated HNO<sub>3</sub> to the specified volumes with reagent water for initial calibration.
    - 10.2.4.1.1. Each target analyte in the ICV solution must be at a concentration within the established linear dynamic range.
  - 10.2.4.2. Use the analyte and acid concentrations outlined in Appendix C as guidance to prepare the ICV solutions ***if the commercially prepared custom reference standard solutions are unavailable.***
  - 10.2.4.3. The ICV solution must be of a source differing from that used for the initial one-point calibration. If it is of the same source, then it must be of different lot.
- 10.2.5. Continuing Calibration Verification (CCV) Solutions
  - 10.2.5.1. Dilute the appropriate volumes of the initial calibration standards with equal volume of calibration blank.
    - 10.2.5.1.1. Each target analyte in the CCV solution is at a concentration near the midpoint of the calibration curve.
  - 10.2.5.2. Use the analyte and acid concentrations outlined in Appendix C as guidance to prepare the CCV solutions ***if the commercially prepared custom reference standard solutions are unavailable.***
  - 10.2.5.3. Prepare the CCV solution fresh daily.
  - 10.2.5.4. The CCV solution is of a source same as that used for the initial one-point calibration.
- 10.2.6. Internal Standard Solution



STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 14 of 70

- 10.2.6.1. Prepare the internal standard solution by diluting the appropriate volumes of the stock standards, concentrated HCl, and concentrated HNO<sub>3</sub> to 2000 mL with reagent water.
- 10.2.6.2. Use the analyte and acid concentrations outlined in Appendix C as guidance to prepare the internal standard solution **if the commercially prepared custom reference standard solutions are unavailable.**
- 10.2.6.3. The internal standard solution contains 5 ppm each of Ho, Tb, and Y. It is used to reduce or overcome interferences (see Section 7.2. and Section 7.3.).
- 10.2.7. Potential Interference Check Solution
  - 10.2.7.1. Dilute the appropriate volumes of the stock standards, concentrated HCl, and concentrated HNO<sub>3</sub> to the desired volumes with reagent water for potential interference check.
  - 10.2.7.2. The potential interference check solution contains 200 ppm each of Al, Ca, Cr, Cu, Fe, Mg, Mn, Tl, and V. It is used to establish the potential interference table (see Section 12.1.).
- 10.2.8. Daily Spectral Interference Check Solutions (ICS-AB and ICS-A)
  - 10.2.8.1. Dilute the appropriate volumes of the stock standards, concentrated HCl, and concentrated HNO<sub>3</sub> to the specified volumes with reagent water for daily spectral interference check.
  - 10.2.8.2. Use the analyte and acid concentrations outlined in Appendix C as guidance to prepare the ICS-AB and ICS-A solutions **if the commercially prepared custom reference standard solutions are unavailable.**
  - 10.2.8.3. The ICS-AB and ICS-A solutions contain known concentrations of interfering elements. They are used to verify the interelement correction factors.
- 10.2.9. Spike Standard Solutions
  - 10.2.9.1. Prepare the spike working standard solutions by diluting the appropriate volumes of the second source stock standards, concentrated HCl, and concentrated HNO<sub>3</sub> to the specified volumes with reagent water.
  - 10.2.9.2. Use the analyte and acid concentrations outlined in Appendix C as guidance to prepare the spike working standard solutions **if the commercially prepared custom reference standard solutions are unavailable.**
  - 10.2.9.3. The spike standard solutions are used to prepare QC check samples such as laboratory control samples (LCS/LCSDs), matrix spikes (MS/MSDs), and post digestion spikes (PDSs).

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 15 of 70

- 10.2.9.4. The spike standard must be of a source differing from that used for the initial one-point calibration. If it is of the same source, then it must be of different lot.
- 10.2.9.5. Add 250 µL of the spike standard to each 50 mL aliquot of aqueous LCS/LCSD and MS/MSD sample prior to digestion.
- 10.2.9.6. Add 500 µL of the spike standard to each 2 g aliquot of solid LCS/LCSD and MS/MSD sample prior to digestion.
- 10.2.9.7. Add 50 µL of the spike standard to each 10 mL aliquot of PDS sample after digestion.
- 10.2.9.8. Add 250 µL of the spike standard to each 5 mL aliquot of mobility-procedure extract designated as LCS/LCSD and MS/MSD prior to dilution and acidification.
- 10.2.10. Linear Dynamic Range Solutions
  - 10.2.10.1. Prepare a minimum of three different concentrations of the linear dynamic range solutions in the same acid matrix by diluting the spike standard solutions or the stock standard solutions. The analyst determines the applicable concentrations.
  - 10.2.10.2. The linear dynamic range solutions contain various concentrations of compatible elements. They are used to establish linear dynamic range (see Section 12.6.).
- 10.2.11. All working standards must be replaced after the stock standard expiration dates (unless specified otherwise) or sooner if routine QC or comparison with check standards indicates a problem.
  - 10.2.11.1. Add the appropriate types and volumes of acids such that the mixed standard solutions are matrix matched with the sample digestates.
    - 10.2.11.1.1. If internal standards are utilized, then the types and volumes of acids added to a mixed standard solution do not need to be matrix matched with the sample digestates.
  - 10.2.11.2. Care should be taken when preparing the mixed standards to ensure that the elements are compatible and stable together.
  - 10.2.11.3. Transfer the mixed standard solutions to FEP fluorocarbon or previously unused polyethylene or polypropylene bottles for storage.
  - 10.2.11.4. Demonstrate the stability of a low-level working standard (i.e., concentration < 1 ppm) prior to use.
- 10.2.12. All stock standards must be inspected and documented in the Chemicals and Supplies Verification Logbook prior to use.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 16 of 70

## 11. SAMPLE COLLECTION, PRESERVATION, CONTAINERS AND HOLDING TIMES

- 11.1. Aqueous samples should be collected in 250 mL pre-cleaned high density polyethylene (HDPE) containers with Teflon-lined closures.
  - 11.1.1. Aqueous samples for dissolved metals determination shall be field filtered within 15 minutes of sample collection and preserved with 1:1 HNO<sub>3</sub> solution to pH < 2.
  - 11.1.2. Aqueous samples for total metals determination shall be preserved with 1:1 HNO<sub>3</sub> solution to pH < 2.
- 11.2. Solid samples should be collected in 4 oz or 8 oz pre-cleaned clear glass wide-mouth jars with Teflon-lined closures, or 6 in decontaminated brass or stainless steel sleeves with Teflon-lined closures.
- 11.3. Mobility-procedure extracts should be collected in 3 oz pre-cleaned polypropylene digestion tubes with polypropylene lids, or 250 mL pre-cleaned HDPE containers with Teflon-lined closures.
  - 11.3.1. Mobility-procedure extracts shall be preserved with 1:1 HNO<sub>3</sub> solution to pH < 2.
  - 11.3.2. If precipitate is observed upon the addition of 1:1 HNO<sub>3</sub> solution to a small aliquot of the mobility-procedure extract, do not acid preserve the mobility-procedure extract. Digest the mobility-procedure extract within 24 hours.
- 11.4. Aqueous and Solid samples shall be maintained in a chilled state (0–6°C), not frozen, post sample collection until received at the laboratory.
- 11.5. Upon receipt, the aqueous and solid samples are stored in a 0–6°C cooler.
  - 11.5.1. Unfiltered aqueous samples for dissolved metals determination must be filtered as soon as possible, immediately preserved with 1:1 HNO<sub>3</sub> solution to pH < 2, and digested and/or analyzed within 6 months of sample collection.
  - 11.5.2. Filtered aqueous samples with acid preservation (pH < 2) for dissolved metals determination must be digested and/or analyzed within 6 months of sample collection.
  - 11.5.3. Filtered aqueous samples without acid preservation (pH ≥ 2) for dissolved metals determination must be preserved with 1:1 HNO<sub>3</sub> solution to pH < 2 for at least 24 hours prior to digestion or analysis, and digested and/or analyzed within 6 months of sample collection.
  - 11.5.4. Aqueous samples with acid preservation (pH < 2) for total metals determination must be digested and analyzed within 6 months of sample collection.
  - 11.5.5. Aqueous samples without acid preservation (pH ≥ 2) for total metals determination must be preserved with 1:1 HNO<sub>3</sub> solution to pH < 2 for at least 24 hours prior to digestion, and digested and analyzed within 6 months of sample collection.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 17 of 70

- 11.5.6. Solid samples must be digested and analyzed within 6 months of sample collection.
- 11.5.7. Mobility-procedure extracts with acid preservation ( $\text{pH} < 2$ ) must be digested and analyzed within 180 days post mobility extraction.
  - 11.5.7.1. Mobility-procedure extracts shall be stored at ambient temperature prior to digestion and analysis.
- 11.5.8. Mobility-procedure extracts without acid preservation ( $\text{pH} \geq 2$ ) must be preserved with 1:1  $\text{HNO}_3$  solution to  $\text{pH} < 2$  for at least 24 hours prior to digestion, and digested and analyzed within 180 days post mobility extraction.
  - 11.5.8.1. Mobility-procedure extracts shall be stored at ambient temperature prior to digestion and analysis.
- 11.6. Refer to the specific SOPs of the preparatory methods and Appendix D for additional information on sample collection, preservation, and holding time.

## 12. QUALITY CONTROL

### 12.1. Potential Interference Table

- 12.1.1. Following the initial instrument setup, the potential interference table (see Appendix B) must be established prior to initial calibration.
  - 12.1.1.1. The potential interference table is established by analyzing the potential interference check solution (see Section 10.2.7.).
- 12.1.2. The potential interference table should be updated every six months, when the daily spectral interference check is deemed unacceptable, or when an instrumentation change occurs.

### 12.2. Instrument Detection Limit (IDL)

- 12.2.1. The instrument detection limit for each analyte shall be performed at initial instrument setup.
  - 12.2.1.1. The IDL in mg/L is determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a method blank with seven consecutive measurements per day.
- 12.2.2. The data and calculations should be kept on file.

### 12.3. Initial Calibration (IC)

- 12.3.1. The initial one-point calibration must be established daily prior to the processing of sample digestates.
  - 12.3.1.1. The calibration curve is established with one calibration blank and one high-level calibration standard.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 18 of 70

- 12.3.1.2. The concentration level of each analyte in the high-level calibration standard shall not exceed its anticipated linear dynamic range.
- 12.3.2. The IC is deemed valid if the replicate %RSD for each analyte is  $\leq 5\%$ .
- 12.3.3. If these criteria are not met, then the calibration is unacceptable for sample analysis to begin. Effect corrective action and recalibrate.
- 12.4. Initial Calibration Verification (ICV)
  - 12.4.1. Immediately following the establishment of a valid initial calibration, an ICV standard must be analyzed prior to sample analysis.
  - 12.4.2. The initial calibration is deemed valid if the replicate %RSD for each analyte is  $\leq 5\%$ , and the %D for each analyte is  $\leq 10\%$ .
  - 12.4.3. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to begin. An unacceptable ICV result indicates either a disagreement between like solutions from separate sources or a change in instrument conditions. Normally, this is caused when at least one of the solutions is no longer intact (representative of the stated concentration). Document the unacceptable result and re-analyze the ICV within 2 hours after the failed ICV. If the ICV remains unacceptable, investigate, effect corrective action, which may include re-preparation of standard solutions or instrument maintenance, and recalibrate.
- 12.5. Initial Calibration Blank (ICB)
  - 12.5.1. Immediately following the analysis of an ICV standard, an ICB must be analyzed prior to sample analysis.
  - 12.5.2. The instrument operating condition is deemed satisfactory for sample analysis to begin if no analytes are detected at a concentration  $\geq$  RL (or the limit specified in the project specific DQO).
  - 12.5.3. If these criteria are not met, no sample analysis shall begin. Determine the source of contamination. Re-prepare and re-analyze the ICB.
- 12.6. Linear Dynamic Range
  - 12.6.1. Following the initial instrument setup, the upper limit of the linear dynamic range for each analyte must be established for each wavelength utilized prior to initial calibration.
    - 12.6.1.1. The upper range limit is established for each wavelength by determining the signal responses from a minimum of three, preferably five, different concentration standards across the range.
      - 12.6.1.1.1. The concentration level of each analyte in the lowest concentration standard shall be at or below the RL.
    - 12.6.1.2. The ranges which may be used for the analysis of samples should be judged by the analyst from the resulting data. The

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 19 of 70

data, calculations and rationale for the choice of range made should be documented and kept on file.

- 12.6.2. Following the establishment of a valid initial calibration, the upper range limit must be checked every six months, and a new upper range limit should be determined whenever there is a significant change in instrument response.

12.6.2.1. The analyst should be aware that if an analyte that is present above its upper range limit is used to apply an interelement correction, the correction may not be valid and those analytes where the interelement correction has been applied may be inaccurately reported.

- 12.6.3. The upper range limit is deemed valid if the %D for each analyte in a high-level check standard analyzed and quantitated against the calibration curve is  $\leq 10\%$ .

- 12.6.4. Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. Hence, non-linear second order curve may be used if the instrument allows it.

12.6.4.1. The effective range must be checked, and the correlation coefficient of the second order curve fit should be  $\geq 0.995$ .

12.6.4.2. Non-linear response curves should be revalidated and recalculated every six months. These curves are much more sensitive to changes in operating conditions than the linear lines and should be checked whenever there have been moderate equipment changes.

## 12.7. Daily Spectral Interference Check (ICS-AB and ICS-A)

- 12.7.1. Following the establishment of a valid initial calibration, an ICS-AB and ICS-A solutions must be analyzed daily prior to sample analysis. Per client request or project specific DQOs, an ICS-AB solution must be analyzed at the end of sequence.

12.7.1.1. The daily spectral interference check solutions are utilized to verify either the accuracy of the interelement correction factors if interelement corrections are applied, or the absence of interferences if interelement corrections are not applied.

- 12.7.2. The ICS-AB is deemed acceptable if the %D for each analyte is  $\leq 20\%$ .

- 12.7.3. The ICS-A is deemed acceptable if the absolute value of the concentration for each non-spiked analyte is  $< RL$  (unless it is a verified trace impurity from one of the spiked analytes).

- 12.7.4. If these criteria are not met, no sample analysis shall begin. Determine the source of problem, effect corrective action, and re-analyze the ICS-AB and/or ICS-A.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 20 of 70

- 12.7.4.1. If an ICS-AB and/or ICS-A at the start of sequence are unacceptable, effect corrective action prior to analyzing any samples.
  - 12.7.4.2. Per client request or project specific DQOs, if an ICS-AB at the end of sequence is unacceptable, effect corrective action and re-analyze all samples since the last acceptable ICS-AB.
  - 12.7.5. All interelement spectral correction factors or multivariate correction matrices must be verified and updated every six months, when the daily spectral interference check is deemed unacceptable, or when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions, occurs.
  - 12.8. Continuing Calibration Verification (CCV)
    - 12.8.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily prior to sample analysis, after every batch of 10 samples or portion thereof within a 24-hour shift, and at the end of sequence.
    - 12.8.2. The initial calibration is deemed valid if the replicate %RSD for each analyte is  $\leq 5\%$ , and the %D for each analyte is  $\leq 10\%$ .
    - 12.8.3. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to resume. Document the unacceptable result and re-analyze the CCV within 2 hours after the failed CCV. If the CCV remains unacceptable, effect corrective action and recalibrate.
      - 12.8.3.1. If a failed CCV is the first of the day, effect corrective action and recalibrate prior to analyzing any samples.
      - 12.8.3.2. If a failed CCV is not the first of the day, effect corrective action, recalibrate, and re-analyze all samples since the last acceptable CCV.
  - 12.9. Continuing Calibration Blank (CCB)
    - 12.9.1. Immediately following the analysis of a CCV standard, a CCB must be analyzed prior to sample analysis.
    - 12.9.2. The instrument operating condition is deemed satisfactory for sample analysis to resume if no analytes are detected at a concentration  $\geq$  RL (or the limit specified in the project specific DQO).
    - 12.9.3. If these criteria are not met, no sample analysis shall resume. Determine the source of contamination. Re-prepare and re-analyze the CCB.
      - 12.9.3.1. If a failed CCB is the first of the day, effect corrective action prior to analyzing any samples.
      - 12.9.3.2. If a failed CCB is not the first of the day, effect corrective action and re-analyze all samples since the last acceptable CCB.
  - 12.10. Event Based Quality Control (MBs and LCS/LCSDs)
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STANDARD OPERATING PROCEDURE  
Title: EPA 8010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 21 of 70

- 12.10.1. Event based quality control consists of QC samples prepared and processed with each preparatory event. This consists of a method blank (MB), a laboratory control sample (LCS), and a laboratory control sample duplicate (LCSD).
- 12.10.1.1. LCSD shall be prepared and processed if there is insufficient sample amount to perform matrix based QC (i.e., MS/MSD), or if it is mandatory per client request or project specific DQOs.
- 12.10.2. The acceptance criteria for MBs are as follows:
- 12.10.2.1. Ideally, the concentrations of target analytes in an MB should be less than the respective limits specified in the project specific DQO. In the absence of project specific DQO, the concentrations of target analytes in an MB should be less than or equal to the respective RLs. If regulatory limits are available, the concentrations of target analytes in an MB should be less than 10% of the respective regulatory limits. If the concentration of any target analyte exceeds its specified limit, the source of contamination must be investigated and, if possible, eliminated.
- 12.10.2.2. If a target analyte is found in the MB, but not in the associated samples, report the sample and MB data without qualification.
- 12.10.2.3. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-processed and/or re-analyzed.
- 12.10.3. The acceptance criteria for LCS/LCSD elements are as follows:
- 12.10.3.1. The lower and upper acceptance limits for %REC of each LCS/LCSD element are 80% and 120%, respectively. The RPD is  $\leq 20\%$  (between LCS and LCSD).
- 12.10.3.1.1. If historical data is available, the lower and upper acceptance limits for %REC and RPD of each LCS/LCSD element are based upon the historical average recovery  $\pm 3S$  that is updated at least annually.
- 12.10.3.2. All LCS (including LCSD if required) elements must be within acceptance limits. However, if a large number of analytes are in the LCS, it becomes statistically likely that a few will be outside of control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary. Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary.



STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 22 of 70

- 12.10.3.3. ME is defined as being beyond the LCS control limit (3 standard deviations), but within the ME limits. ME limits are between 3 and 4 standard deviations around the mean.
- 12.10.3.4. The number of allowable marginal exceedances is based on the number of analytes in the LCS. If more analytes exceed the LCS control limits than is allowed, or if any one analyte exceeds the ME limits, the LCS fails and corrective action is necessary. This marginal exceedance approach is relevant for methods with long lists of analytes. It will not apply to target analyte lists with fewer than 11 analytes.
- 12.10.3.5. The number of allowable marginal exceedances is as follows:

Number of Analytes in LCS	Number of Analytes Allowed in ME of the LCS Control Limit
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
< 11	0

- 12.10.3.6. Marginal exceedances must be random. If the same analyte exceeds the LCS control limit 2 out of 3 consecutive LCS, it is an indication of a systemic problem. The source of the error must be located and corrective action taken.
- 12.10.3.7. If the problem was not related to the digestion process, then the LCS/LCSD and all associated sample digestates must be re-analyzed. If the failure was associated with the digestion process, then all associated samples must be re-processed and re-analyzed.

#### 12.11. Matrix Based Quality Control (MS/MSDs)

- 12.11.1. Matrix based quality control consists of QC samples prepared and processed using actual environmental samples. This consists of a matrix spike (MS) and matrix spike duplicate (MSD).
- 12.11.2. The acceptance criteria for MS/MSD elements are as follows:
- 12.11.2.1. The lower and upper acceptance limits for %REC of each MS/MSD element are 75% and 125%, respectively. The RPD is  $\leq 20\%$  (between MS and MSD).
- 12.11.2.1.1. If historical data is available, then the lower and upper acceptance limits for %REC and %RPD of each MS/MSD element are based upon the historical average recovery  $\pm 3S$  that is updated at least annually.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 23 of 70

- 12.11.2.2. When the %REC and RPD of the MS/MSD elements are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.
- 12.11.2.3. If the %REC and/or RPD of the MS/MSD elements are not within the established acceptance limits, the analytical system performance shall be suspect.
- 12.11.3. Unacceptable %REC values are typically caused by matrix effects or poor instrument performance/technique. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.
- 12.12. If the %REC or RPD of the LCS/LCSD and MS/MSD are unacceptable, all associated sample data must be invalidated and all associated samples re-processed and re-analyzed.
- 12.13. Dilution Test
- 12.13.1. If the analyte concentration is sufficiently high (minimally, a factor of 10 above the instrument detection limit after dilution), an analysis of a 1:5 dilution should agree within  $\pm 10\%$  of the original determination.
- 12.13.2. If this criterion is not met, a physical or chemical interference effect shall be suspect. Perform post digestion spike addition.
- 12.14. Post Digestion Spike (PDS) Addition
- 12.14.1. A PDS sample is prepared by adding the spike standard to a portion of a digested sample, or its dilution. The spike addition should produce a concentration of 10–100 times the RL.
- 12.14.2. The lower and upper acceptance limits for %REC of each PDS element are 75% and 125%, respectively.
- 12.14.3. If the %REC of the PDS element is not within the established acceptance limits, a matrix effect shall be suspect. Perform MSA (see Section 14.15.) on all samples in the same preparation batch per client request or project specific DQOs.
- 12.15. Additional information regarding internal quality control checks is provided in SOP-T020.

### 13. CALIBRATION AND STANDARDIZATION

#### 13.1. Initial Demonstration of Performance

- 13.1.1. Document the selection criteria for background correction points; analytical dynamic ranges, the applicable equations, and the upper limits of those

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 24 of 70

ranges; the method and instrument detection limits; and the determination and verification of interelement correction equations or other routines for correcting spectral interferences.

13.1.2. Generate the data using the same instrument, operating conditions, and calibration routine to be used for sample analysis.

13.1.3. Keep the data on file and available for review.

#### 13.2. Pipetter

13.2.1. Calibrate the pipetter according to the procedure outlined in the current revision of SOP-T043.

#### 13.3. Spectrometer Initial Calibration

13.3.1. Establish an acceptable one-point calibration curve. The acceptance criteria for the initial calibration are listed in Section 12.3.

13.3.2. After obtaining an acceptable one-point calibration curve and prior to processing field or QC sample digestates, an ICV standard and ICB must be analyzed to verify the initial calibration. The acceptance criteria for the ICV and ICB are listed in Section 12.4. and Section 12.5.

13.3.3. The initial one-point calibration and ICV shall include all anticipated target analytes for the duration of the use of the initial calibration.

### 14. PROCEDURE

#### 14.1. Instrument Setup

14.1.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable (usually requiring at least 30 minutes of operation) prior to calibration. Follow the instructions provided by the instrument manufacturer for operating conditions.

14.1.1.1. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality.

14.1.1.2. Deviations from instructions provided by the instrument manufacturer must be documented and approved by the Group Leader.

14.1.1.3. Use the following ICP-AES operating conditions as guidance.

Description	Operating Condition
RF Power	1100~1450 watts
Viewing Height	
Axial Plasma	14~18 mm
Radial Plasma	~1~5 mm
Argon Coolant Flow	15~19 L/min
Argon Nebulizer Flow	0.5~1.5 L/min

STANDARD OPERATING PROCEDURE  
Title: EPA 8010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 25 of 70

Description (Cont.)	Operating Condition
Sampler	
Pump rate	0.6~1.0 mL/min
Wash time	15 sec
Preflush Time	1 min
Read Time	2~10 sec
Read Delay Time	15 sec
Number of readings/replicate	2

- 14.1.1.4. Repeatable interference correction factors can be achieved by adjusting the argon aerosol flow to reproduce the Cu/Mn intensity ratio at 324.754 nm and 257.610 nm respectively.
- 14.1.2. Refer to Appendix A for specific wavelengths. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference.
- 14.1.3. Optimize the plasma operating conditions prior to the use of the instrument. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure. This routine is not required on a daily basis, but only is required when first setting up a new instrument, or following a change in operating conditions. Follow the instrument manufacturer's instructions to optimize the plasma operating conditions.
- 14.1.3.1. Ignite the radial plasma and select an appropriate incident RF power. Allow the instrument to become thermally stable (about 30 to 60 minutes of operation). While aspirating a 1000 µg/L solution of yttrium, follow the instrument manufacturer's instructions and adjust the aerosol carrier gas flow rate through the nebulizer so a definitive blue emission region of the plasma extends approximately from 5 to 20 mm above the top of the load coil. Record the nebulizer gas flow rate or pressure setting for future reference. The yttrium solution can also be used for coarse optical alignment of the torch by observing the overlay of the blue light over the entrance slit to the optical system.
- 14.1.3.2. After establishing the nebulizer gas flow rate, determine the solution uptake rate of the nebulizer in mL/min by aspirating a known volume of a calibration blank for a period of at least three minutes. Divide the volume aspirated by the time in minutes and record the uptake rate. Set the peristaltic pump to deliver that rate in a steady even flow.
- 14.1.3.3. Profile the instrument to align it optically as it will be used during analysis. Follow the instrument manufacturer's instructions for optimization in the axial and radial modes.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 26 of 70

- 14.1.4. Align the torch viewing position weekly.
  - 14.1.4.1. Aspirate a standard solution containing 10 mg/L of Mn for radial viewing and 1 mg/L of Mn for axial viewing.
  - 14.1.4.2. Allow the instrument software to set the torch viewing position with the highest signal intensity.
  - 14.1.4.3. For radial viewing, the intensity of Mn at 257.610 nm should be greater than 10000 counts. For axial viewing, the intensity of Mn at 257.610 nm should be greater than 700000 counts.
  - 14.1.4.4. If these criteria are not met, effect corrective action and re-align the torch viewing position.
- 14.1.5. Check the sensitivity daily.
  - 14.1.5.1. Aspirate a standard solution containing 7.5 mg/L of As and 7.5 mg/L of Pb.
  - 14.1.5.2. For axial viewing, the standard emission count of As at 193.696 nm should be greater than 10000, and the standard emission count of Pb at 220.353 nm should be greater than 40000.
  - 14.1.5.3. If these criteria are not met, perform instrument maintenance, re-align the torch viewing position, and check the sensitivity prior to initial calibration.
- 14.1.6. The instrument operating condition finally selected as being optimum should provide the lowest reliable instrument detection limits (IDLs).
- 14.1.7. If either the instrument operating conditions (such as incident power or nebulizer gas flow rate) are changed, or a new torch injector tube with a different orifice internal diameter is installed, then the plasma and viewing height should be re-optimized.
- 14.1.8. After completing the initial optimization of operating conditions, and before analyzing samples, an interelement spectral interference correction routine to be used for sample analysis must be established and initially verified.
  - 14.1.8.1. A general description of spectral interferences and the analytical requirements for background correction are discussed in Section 7.
  - 14.1.8.2. The criterion for determining the presence of an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls beyond  $\pm$  one reporting limit from zero. The upper control limit is the analyte instrument detection limit.
  - 14.1.8.3. Once established, the entire routine must be verified every six months. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verifications of the routine should be kept on file.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 27 of 70

- 14.1.9. Before daily calibration, and after the instrument warm-up period, the nebulizer gas flow rate must be reset to the determined optimized flow. If a mass flow controller is being used, it should be set to the recorded optimized flow rate. In order to maintain valid spectral interelement correction routines, the nebulizer gas flow rate should be the same (< 2% change) from day to day.
- 14.1.10. For operation with organic solvents, the use of the auxiliary argon inlet is recommended, as is the use of solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power, to obtain stable operation and precise measurements.
- 14.1.11. Program the system to average a minimum of two integrations (i.e., replicate readings) on each blank, standard, and sample. Report the average.
  - 14.1.11.1. If the %RSD for an analyte in a standard is > 5%, re-analyze the standard. If the %RSD criterion remains unacceptable, investigate, effect corrective action, which may include re-preparation of the standard solution, and recalibrate, if necessary.
  - 14.1.11.2. If the %RSD for an analyte in a sample is > 20%, and the analyte concentration exceeds its RL, re-analyze the sample. If the %RSD criterion remains unacceptable, investigate and effect corrective action.
- 14.2. Establish sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.
  - 14.2.1. Establish method detection limits (MDLs) for all wavelengths utilized for each type of matrix analyzed and for each preparation method used and for each instrument. Additional information regarding determination of detection limits is provided in SOP-T006.
  - 14.2.2. Establish the upper limit of the linear dynamic range for each wavelength utilized (see Section 12.6.).
  - 14.2.3. Verify that the instrument configuration and operating conditions satisfy the analytical requirements, and maintain quality control data confirming instrument performance and analytical results.
- 14.3. Establish a calibration curve to cover the appropriate concentration range (see Section 13.3.).
- 14.4. Following the establishment of a valid initial calibration, an ICS-AB and ICS-A solutions must be analyzed daily prior to sample analysis. Per client request or project specific DQOs, an ICS-AB solution must be analyzed at the end of sequence. The acceptance criteria are listed in Section 12.7.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 28 of 70

- 14.4.1. If an ICS-AB and/or ICS-A at the start of sequence are unacceptable, effect corrective action prior to analyzing any samples.
- 14.4.2. Per client request or project specific DQOs, if an ICS-AB at the end of sequence is unacceptable, effect corrective action and re-analyze all samples since the last acceptable ICS-AB.
- 14.5. Following the establishment of a valid initial calibration, a CCV standard and CCB must be analyzed daily prior to sample analysis, after every batch of 10 samples or portion thereof within a 24-hour shift, and at the end of sequence. If the QC criteria are met, the initial calibration is assumed to be valid and sample analysis may resume. The acceptance criteria are listed in Section 12.8. and Section 12.9.
  - 14.5.1. If a failed CCV/CCB is the first of the day, effect corrective action and recalibrate prior to analyzing any samples.
  - 14.5.2. If a failed CCV/CCB is not the first of the day, effect corrective action, recalibrate, and re-analyze all samples since the last acceptable CCV/CCB.
- 14.6. Following digestion by one of the methods specified in Section 5.3., the digestates for the QC and actual environmental samples are received in digestion tubes. After transferring aliquots of the digestates to autosampler vessels, the autosampler vessels are then loaded onto the ICP-AES sample tray.
  - 14.6.1. Preliminary treatment of most matrices is necessary due to the complexity and variability of sample matrices.
  - 14.6.2. Acid digestion is not necessary if groundwater samples for dissolved metals determination are prefiltered and acidified prior to analysis.
    - 14.6.2.1. All associated QC samples (i.e., MB, LCS/LCSD, MS/MSD, and PDS) in the same preparation batch must undergo the same filtration and acidification procedures.
    - 14.6.2.2. Samples which are not digested must either use an internal standard or be matrix-matched with the standards.
- 14.7. Blank, standard, and sample vessels are loaded in the following or other logical order:
  - 1) Calibration Blank (CB)
  - 2) Initial Calibration Standard
  - 3) Initial Calibration Verification (ICV)
  - 4) Initial Calibration Blank (ICB)
  - 5) Interference Check Solution AB (ICS-AB)
  - 6) Interference Check Solution A (ICS-A)
  - 7) Continuing Calibration Verification (CCV)
  - 8) Continuing Calibration Blank (CCB)
  - 9) Method Blank (MB)
  - 10) Laboratory Control Samples (LCS)
  - 11) Laboratory Control Sample Duplicates (LCSD)
  - 12) Samples (up to 10 per batch, including QC check samples and MBs)
  - 13) Matrix Spike (MS)

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 29 of 70

- 14) Matrix Spike Duplicate (MSD)
  - 15) Dilution Test Sample (per client request or project specific DQOs)
  - 16) Post Digestion Spike (PDS) (per client request or project specific DQOs)
  - 17) Ending ICS-AB (per client request or project specific DQOs)
  - 18) Ending CCV
  - 19) Ending CCB
- 14.7.1. Item 1: The CB is an aliquot of acidified reagent water used to establish the zero point of the initial calibration curve.
- 14.7.2. Item 2: The initial calibration standard is a high-level calibration standard used to establish the initial calibration curve.
- 14.7.3. Item 3: The ICV is a second source standard used to verify the acceptance of the initial one-point calibration. An acceptable ICV is required immediately following initial calibration.
- 14.7.4. Item 4: The ICB is an aliquot of acidified reagent water used to monitor contamination. An acceptable ICB is required immediately following ICV.
- 14.7.5. Items 5, 6, and 17: The ICS-AB and ICS-A are used to verify the accuracy of the interelement correction factors. An acceptable ICS-AB and ICS-A are required daily prior to sample analysis. Per client request or project specific DQOs, an acceptable ICS-AB is required at the end of sequence.
- 14.7.6. Items 7 and 18: A CCV is a standard used to verify the acceptance of the initial one-point calibration on a continuing basis. An acceptable CCV is required daily prior to sample analysis, after every batch of 10 samples or portion thereof within a 24-hour shift, and at the end of sequence.
- 14.7.7. Items 8 and 19: A CCB is an aliquot of acidified reagent water used to monitor contamination. An acceptable CCB is required immediately following CCV.
- 14.7.8. Item 9: The MB is a known matrix similar to the samples being analyzed which is processed concurrently with the associated samples. In the processing of the MB, reagents and procedures identical to those for actual samples are used.
- 14.7.8.1. For aqueous samples, the MB consists of clean reagent water. For solid samples, the MB consists of clean Teflon chips (or glass beads). For mobility-procedure extracts, the MB consists of the mobility-procedure extract designated as MB.
  - 14.7.8.2. One MB is required every day preparatory methods (i.e., leachings, filtrations, digestions, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.
  - 14.7.8.3. When samples that are processed together are analyzed on separate instruments or on separate analytical shifts, the MB associated with those samples must be analyzed on at least one



STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 30 of 70

of the instruments. A solvent blank consisting of acidified reagent water must be analyzed on all other instruments where the associated samples are analyzed to demonstrate that the instruments are not contributing contaminants to the samples.

- 14.7.9. Item 10: The LCS is a known matrix which has been spiked with known concentrations of specific target analytes. The purpose of the LCS is to demonstrate that the entire analytical process and systems are in control. The LCS is processed concurrently with the associated samples. In the processing of the LCS, reagents and procedures identical to those for actual samples are used.
- 14.7.9.1. For aqueous samples, the LCS consists of the specified elements spiked into clean reagent water. For solid samples, the LCS consists of the specified elements spiked into clean Teflon chips (or glass beads). For mobility-procedure extracts, the LCS consists of the specified elements spiked into the mobility-procedure extract designated as LCS.
- 14.7.9.2. One LCS is required every day preparatory methods (i.e., leachings, filtrations, digestions, etc.) are performed for every batch of 20 samples per matrix or portion thereof, whichever is more frequent.
- 14.7.10. Item 11: The LCSD is handled identically to the LCS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the LCS in combination with the LCSD can be used to assess the precision of the analytical process. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.5.
- 14.7.10.1. LCSD is processed and analyzed if there is insufficient sample amount to perform matrix based QC (i.e., MS/MSD), or if it is mandatory per client request or project specific data quality objectives (DQOs).
- 14.7.11. Item 12: Up to 10 sample (including QC check sample and method blank) digestates per batch. Digestates should be sufficiently diluted if concentrations exceed the calibration range. Dilution of digestates will result in increased reporting limits.
- 14.7.11.1. All dilutions should keep the responses in the upper half of the linear range of the curve.
- 14.7.11.2. Digestates with concentrations exceeding the calibration range but within the linear dynamic range may be reported without dilution per client request or project specific DQOs.
- 14.7.12. Item 13: The MS is the actual sample matrix spiked with known concentrations of specific target analytes. The sample which is spiked for the MS is processed concurrently with the associated samples. In the

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 31 of 70

processing of the MS, reagents and procedures identical to those for actual samples are used.

14.7.12.1. The purpose of the MS is to assess the effect of a sample matrix on the recovery of target analytes (i.e., assess the accuracy of the analytical measurements of the matrix). The measurement is expressed as percent recovery (%REC). The formula for calculating %REC is listed in Section 15.4.

14.7.12.2. One MS is required for every batch of 20 samples per matrix or portion thereof processed concurrently.

14.7.13. Item 14: The MSD is handled identically to the MS discussed in the previous section. In addition to assessing the accuracy of the analytical measurement, the MS in combination with the MSD can be used to assess the precision of the analytical measurements. The measurement is expressed as relative percent difference (RPD). The formula for calculating RPD is listed in Section 15.5.

14.7.14. Item 15: The dilution test sample is prepared from the five-fold dilution of a high concentration sample post digestion. The high concentration sample is diluted to one-fifth of the original concentration post digestion to confirm that no interference is observed in the original sample.

14.7.14.1. The purpose of the dilution test sample is to assess matrix effects.

14.7.14.2. To comply with client request or project specific DQOs, one dilution test sample is required for every batch of 20 samples per matrix or portion thereof processed concurrently.

14.7.15. Item 16: The PDS is the same sample matrix from which the MS/MSD samples were prepared or from another sample in the same preparation batch, and is spiked with known concentrations of specific target analytes post digestion. The sample which will be spiked for the PDS is processed concurrently with the associated samples. In the processing of the PDS, reagents and procedures identical to those for actual samples are used.

14.7.15.1. The purpose of the PDS is to confirm matrix effects. The measurement is expressed as percent recovery (%REC). The formula for calculating %REC is listed in Section 15.4.

14.7.15.2. The number of PDS required is based upon client request or project specific DQOs.

14.7.16. Rinse blanks or solvent blanks consisting of acidified reagent water may be added elsewhere in the sequence, as necessary (i.e., after suspected high concentration sample digestates), to rinse the analytical system or check for potential carryover or cross-contamination.

14.7.16.1. The rinse time is set to one minute. Rinse time may be reduced through a suitable demonstration.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 32 of 70

- 14.8. Ensure that a sufficient amount of internal standard solution is present in the standard vessel at the beginning of the sequence.
- 14.9. Ensure that a sufficient amount of rinse blank is present in the rinse blank bottle, and that a sufficient unused volume exists in the waste container at the beginning of the sequence.
- 14.10. Edit the sequence in the data system. After all correct sample information is entered, save the sequence. After saving the sequence, record pertinent information in the instrument run logbook or on the sequence table printout.
  - 14.10.1. Record the reagent and standard identification numbers on the sequence table printout.
- 14.11. Initiate the sequence.
- 14.12. Dilution test and post digestion spike addition (see Section 12.13. and Section 12.14.) are recommended prior to reporting concentration data for the elements.
  - 14.12.1. It is recommended that these tests be performed with each batch of samples prepared/analyzed to ensure that neither positive nor negative interferences are affecting the measurement of any element or distorting the accuracy of the reported values.
- 14.13. If spectral overlap is suspected, then the use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.
- 14.14. Data Interpretation
  - 14.14.1. Quantitation of a target analyte is based on a reproducible response of the spectrometer within the calibration range and a direct proportionality of the magnitude of response between intensities in the sample digestate and the calibration standard.
    - 14.14.1.1. Proper quantitation requires the appropriate selection of a wavelength from which the intensity can be determined.
    - 14.14.1.2. Determine the concentration based on the initial calibration curve.
      - 14.14.1.2.1. The data system is programmed to perform the calculation of concentration.
    - 14.14.1.3. If the instrument response exceeds the calibration range, dilute the digestate and re-analyze.
- 14.15. Method of Standard Additions (MSA)
  - 14.15.1. The standard addition technique involves adding known amounts of a standard solution to one or more aliquots of a processed sample. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. However, it will not correct for additive interferences which cause a baseline shift.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 33 of 70

- 14.15.1.1. The MSA may be appropriate for analyses of digestates, on analyses submitted as part of a delisting petition, whenever a new sample matrix is being analyzed, and on every batch that fails the post digestion spike addition per client request or project specific DQOs.
- 14.15.2. The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample, each of volume  $V_x$ , are taken. To the first (labeled A) is added a known volume  $V_s$  of a standard analyte solution of concentration  $C_s$ . To the second aliquot (labeled B) is added the same volume  $V_s$  of the acidified reagent water. The analytical signals of A and B,  $S_A$  and  $S_B$ , are measured and corrected for non-analyte signals. The unknown sample concentration  $C_x$  is calculated using the formula listed in Section 15.12.  $V_s$  and  $C_s$  should be chosen so that  $S_A$  is roughly twice  $S_B$  on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.
- 14.15.3. Improved results can be obtained by employing a series of standard additions. A series of standard solutions containing different known quantities of the analyte are added to equal volumes of the sample, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50% of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100% and 150% of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot is shown in Appendix E. A linear regression program may be used to obtain the intercept concentration.
- 14.15.4. For the results of the MSA technique to be valid, the following limitations must be taken into consideration:
- 14.15.4.1. The apparent concentrations from the calibration curve must be linear (correlation coefficient of 0.995 or greater) over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve.
- 14.15.4.2. The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

STANDARD OPERATING PROCEDURE  
Title: EPA 8010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 34 of 70

14.15.4.3. The determination must be free of spectral interference and corrected for nonspecific background interference.

## 15. CALCULATIONS

15.1. The percent relative standard deviation is calculated as follows:

$$\%RSD = \frac{SD}{I_{ave}} \times 100$$

where: %RSD = percent relative standard deviation.  
SD = standard deviation of the intensity readings for the target analyte.  
 $I_{ave}$  = mean of the intensity readings for the target analyte.

15.2. The percent difference of each analyte is calculated as follows:

$$\%D = \frac{|C_{expected} - C_{measured}|}{C_{expected}} \times 100$$

where: %D = percent difference.  
 $C_{expected}$  = concentration of target analyte expected.  
 $C_{measured}$  = concentration of target analyte measured.

Note: Concentrations must be in equivalent units.

15.3. The recovery of each LCS element is calculated as follows:

$$\%REC_{LCS} = \frac{C_{recovered}}{C_{added}} \times 100$$

where: %REC<sub>LCS</sub> = percent recovery of target analyte in LCS (or LCSD).  
 $C_{recovered}$  = concentration of target analyte recovered.  
 $C_{added}$  = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

15.4. The recovery of each MS element is calculated as follows:

$$\%REC_{MS} = \frac{C_{recovered} - C_{sample}}{C_{added}} \times 100$$

where: %REC<sub>MS</sub> = percent recovery of target analyte in MS (or MSD/PDS).  
 $C_{recovered}$  = concentration of target analyte recovered.  
 $C_{sample}$  = concentration of target analyte in environmental sample used.  
 $C_{added}$  = concentration of target analyte added.

Note: Concentrations must be in equivalent units.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 35 of 70

15.5. The relative percent difference is calculated as follows:

$$RPD = \frac{|C_1 - C_2|}{\left(\frac{C_1 + C_2}{2}\right)} \times 100$$

where: RPD = relative percent difference between two measurements ( $C_1$  and  $C_2$ ).

$C_1$  = concentration of target analyte in measurement 1.

$C_2$  = concentration of target analyte in measurement 2.

Note: Concentrations must be in equivalent units.

15.6. The slope and intercept of a linear calibration curve are calculated as follows:

$$M = \frac{I_s - I_b}{C_s - C_b} \quad B = \frac{C_s I_b - C_b I_s}{C_s - C_b}$$

where: M = slope of the calibration curve.

B = intercept of the calibration curve.

$I_s$  = intensity of calibration standard at a specific wavelength.

$I_b$  = intensity of calibration blank at a specific wavelength.

$C_s$  = concentration of calibration standard.

$C_b$  = concentration of calibration blank.

Note: Concentrations must be in equivalent units.

15.7. The target analyte concentration for a sample digestate is calculated as follows:

$$C_x = \frac{I_x - B}{M}$$

where:  $C_x$  = concentration of target analyte in digestate in mg/L.

$I_x$  = intensity of target analyte at a specific wavelength.

B = intercept of the calibration curve.

M = slope of the calibration curve.

15.8. The target analyte concentration for an aqueous sample is calculated as follows:

$$C_A = \frac{C_x \times V_x \times D}{V_A}$$

where:  $C_A$  = concentration of target analyte in aqueous sample in mg/L.

$C_x$  = concentration of target analyte in digestate in mg/L.

$V_x$  = volume of digestate in mL.

$V_A$  = volume of aqueous sample digested in mL.

D = dilution factor, if the sample or digestate was diluted prior to analysis.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 36 of 70

If no dilution was made,  $D = 1$ .

15.9. The target analyte concentration for a solid sample is calculated as follows:

$$C_s = \frac{C_x \times V_x \times D}{W_s}$$

where:  $C_s$  = concentration of target analyte in solid sample in mg/kg.  
 $C_x$  = concentration of target analyte in digestate in mg/L.  
 $V_x$  = volume of digestate in mL.  
 $W_s$  = mass of solid sample digested in g.  
 $D$  = dilution factor, if the digestate was diluted prior to analysis.  
If no dilution was made,  $D = 1$ .

15.10. The target analyte concentration for a solid sample on a dry-weight basis is calculated as follows:

$$C_s = \frac{C_x \times V_x \times D}{W_s \times \left( \frac{C_{ss}}{100} \right)}$$

where:  $C_s$  = concentration of target analyte in solid sample in mg/kg.  
 $C_x$  = concentration of target analyte in digestate in mg/L.  
 $V_x$  = volume of digestate in mL.  
 $W_s$  = mass of solid sample digested in g.  
 $C_{ss}$  = solids content in %.  
 $D$  = dilution factor, if the digestate was diluted prior to analysis.  
If no dilution was made,  $D = 1$ .

15.11. The target analyte concentration for a mobility-procedure extract is calculated as follows:

$$C_{MP} = \frac{C_x \times V_x \times D}{V_{MP}}$$

where:  $C_{MP}$  = concentration of target analyte in mobility-procedure extract in mg/L.  
 $C_x$  = concentration of target analyte in digestate in mg/L.  
 $V_x$  = volume of digestate in mL.  
 $V_{MP}$  = volume of mobility-procedure extract digested in mL.  
Unless specified otherwise,  $V_{MP} = 5$ .  
 $D$  = dilution factor, if the digestate was diluted prior to analysis.  
If no dilution was made,  $D = 1$ .

15.12. The target analyte concentration from single-addition method is calculated as follows:

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 37 of 70

$$C_x = \frac{S_B \times V_s \times C_s}{(S_A - S_B) \times V_x}$$

where:  $C_x$  = concentration of target analyte in sample.  
 $S_A$  = analytical signal (corrected for the blank) of sample aliquot A.  
 $S_B$  = analytical signal (corrected for the blank) of sample aliquot B.  
 $V_s$  = volume of target analyte in standard solution.  
 $C_s$  = concentration of target analyte in standard solution.  
 $V_x$  = volume of target analyte in sample.

Note: Concentrations and volumes must be in equivalent units.

- 15.13. Refer to the preparatory method(s) for additional calculations.
- 15.14. All concentrations shall be reported in mg/L (ppm) for aqueous samples, and mg/kg (ppm) for soil and solid waste samples.
  - 15.14.1. Per client request or project specific DQOs, report all concentrations in mg/kg (ppm) on a dry-weight basis for soil and solid waste samples.
- 15.15. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

## 16. METHOD PERFORMANCE

- 16.1. A demonstration of analytical capability shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.
- 16.2. Calibration protocols specified in Section 13., "Calibration and Standardization," shall be followed.
- 16.3. Proficiency test sample results shall be used to evaluate the ability to produce accurate results.

## 17. POLLUTION PREVENTION

- 17.1. The toxicity, carcinogenicity, and other health hazards associated with the use of most laboratory chemicals have not been precisely defined. Each chemical should be handled assuming it is a potential health hazard.
- 17.2. Exposure to these chemicals should be minimized through the use of proper protective equipment and safe laboratory practices as referenced in the current revision of Eurofins Calscience's Health, Safety, and Respiratory Protection Manual. In general, protective eyewear (e.g. safety glasses or goggles), and protective apparel (e.g. lab coats) and gloves are required to be worn when handling chemicals.
- 17.3. The following additional precautions should be taken, as necessary, when handling high concentrations of hazardous materials:



STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 38 of 70

- 17.3.1. A NIOSH-approved air purifying respirator with cartridges appropriate for the chemical handled.
- 17.3.2. Extended-length protective gloves.
- 17.3.3. Face shield.
- 17.3.4. Full-length laboratory apron.
- 17.4. Processes that promote vaporization of volatile chemicals should be performed in an area well ventilated to the exterior of the laboratory to prevent contamination to other areas in the laboratory.
- 17.5. When working with large amounts of volatile chemicals, the Coordinator must be cautious of the risk of high levels of volatile displacing the atmospheric air within the work area; therefore causing asphyxiation. Air purification respirators are ineffective in this situation and must not be used. The Coordinator must immediately vacate the area until ventilation has effectively reduced the concentration of volatiles. Alternatively, the Coordinator may utilize a self-contained breathing apparatus or other supplied air system if appropriately trained and approved by the Health and Safety Manager.
- 17.6. ► Safety Data Sheets (SDSs) are available for each laboratory standard and reagent chemical. Employees should review and be familiar with the hazards and precautions outlined in the SDS for all chemicals to be used prior to handling.

## 18. DATA ASSESSMENT AND ACCEPTANCE CRITERIA

- 18.1. Ideally, the concentrations of target analytes in an MB should be less than the respective limits specified in the project specific DQO. In the absence of project specific DQO, the concentrations of target analytes in an MB should be less than or equal to the respective RLs. If regulatory limits are available, the concentrations of target analytes in an MB should be less than 10% of the respective regulatory limits. If the concentration of any target analyte exceeds its specified limit, the source of contamination must be investigated and, if possible, eliminated.
  - 18.1.1. If a target analyte is found in the MB but not in the associated samples, report the sample and MB data without qualification.
  - 18.1.2. If a target analyte is found in the MB and in the associated samples, evaluate the analyte in question to determine the effect on the analysis of samples. Determine and eliminate the source of contamination. Professional judgment should be exercised to determine if the data should be qualified or rejected and the samples re-processed and/or re-analyzed.
- 18.2. The acceptance criteria for LCS/LCSD elements are predetermined. The lower and upper acceptance limits for %REC of each LCS/LCSD element are 80% and 120%, respectively. The RPD is  $\leq 20\%$  (between LCS and LCSD). All LCS (including LCSD if required) elements must be within acceptance limits (see Section 12.10.3. for additional information).
  - 18.2.1. Refer to Section 12.10.3.1.1. for acceptance criteria if historical data is available.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 39 of 70

- 18.2.2. If the LCS and/or LCSD %REC is outside of the acceptance limits high, the RPD (when applicable) is within acceptance limits, and all target analytes in the associated samples are not detected, the sample data can be reported without qualification.
  - 18.2.2.1. If the LCS/LCSD is used in place of the MS/MSD due to insufficient sample amount, or if LCS/LCSD is required per client or project specific DQO, both the LCS and LCSD data must be reported.
- 18.3. The acceptance criteria for MS/MSD elements are predetermined. The lower and upper acceptance limits for %REC of each MS/MSD element are 75% and 125%, respectively. The RPD is  $\leq 20\%$  (between MS and MSD).
  - 18.3.1. Refer to Section 12.11.2.1.1. for acceptance criteria if historical data is available.
  - 18.3.2. When the %REC and RPD of the MS/MSD elements are at or within the established acceptance limits, the analytical system is deemed to be compliant with the accuracy and precision requirement of the method for the particular matrix. The MS/MSD data shall be reported with the corresponding sample data.
  - 18.3.3. If the %REC and/or RPD of the MS/MSD elements are not within the established acceptance limits, the analytical system performance shall be suspect.
- 18.4. The acceptance criteria for PDS elements are predetermined. The lower and upper acceptance limits for %REC of each PDS element are 75% and 125%, respectively.
  - 18.4.1. If the %REC of the PDS element and the %REC of the MS/MSD elements are not within the established acceptance limits, matrix effects are confirmed. Perform MSA (see Section 14.15.) on all samples in the same preparation batch per client request or project specific DQOs.
- 18.5. Matrix effects or poor instrument performance/technique typically cause unacceptable %REC values. Unacceptable RPD values are typically caused by sample inhomogeneity or poor instrument performance/technique. To properly evaluate the performance of the analytical system in these situations, refer to the LCS/LCSD. Specifically, an acceptable LCS/LCSD usually supports matrix interference.
- 18.6. Additional information regarding internal quality control checks is provided in SOP-T020.
- 18.7. All concentrations shall be reported in mg/L (ppm) for aqueous samples, and mg/kg (ppm) for soil and solid waste samples.
  - 18.7.1. Per client request or project specific DQOs, report all concentrations in mg/kg (ppm) on a dry-weight basis for soil and solid waste samples.
- 18.8. The data reported shall adhere to the significant figures, rounding, and data reporting procedures outlined in the current revision of SOP-T009.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 40 of 70

## 19. CORRECTIVE ACTIONS

- 19.1. If on the basis of internal or external systems or performance audits, routine monitoring of laboratory support equipment, or QC sample analysis results, analytical systems fail to meet the established criteria, an appropriate corrective action must be implemented.
- 19.2. ►The Operations Director, Project Manager, Quality Control Manager, Group Leader and analyst may be involved in identifying the most appropriate corrective action. If previously reported data are affected or if corrective action will impact the project budget or schedule, the action may directly involve the Laboratory Director.
- 19.3. Corrective actions are generally of two types, immediate and long-term actions.
  - 19.3.1. An **immediate action** is designed to correct or repair nonconforming instruments and measurement systems. The analyst or Group Leader as a result of calibration checks and other QC sample analyses most frequently will identify the need for such an action.
  - 19.3.2. A **long-term action** is designed to eliminate causes of nonconformance. The need for such actions is identified by systems and performance audits. The systematic nonconformances identified during the data generation process and the appropriate corrective measures taken are thoroughly documented in the Corrective Action Record. Examples of this type of action include:
    - 19.3.2.1. Remedial training of staff in technical skills, technique, or implementation of operating procedures.
    - 19.3.2.2. Rescheduling of analytical laboratory routine to ensure analysis within holding times.
    - 19.3.2.3. Revision of standard operating procedures.
    - 19.3.2.4. Replacing personnel, as necessary.
- 19.4. For either type of corrective action, the sequential steps that compose a close-loop corrective action system are as follows:
  - 19.4.1. Define the problem.
  - 19.4.2. Assign responsibility for investigating the problem.
  - 19.4.3. Investigate and determine the cause of the problem.
  - 19.4.4. Assign and accept responsibility for implementing the corrective action.
  - 19.4.5. Determine effectiveness of the corrective action and implement correction.
  - 19.4.6. Verify that the corrective action has eliminated the problem.
- 19.5. Depending on the nature of the problem, the corrective action employed may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be properly documented on a Corrective Action Record.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 41 of 70

## 20. CONTINGENCIES FOR OUT-OF-CONTROL OR UNACCEPTABLE DATA

- 20.1. Out-of-control data are reviewed and verified by the Group Leader of the appropriate department. All samples associated with an unacceptable QC set are then subject to reanalysis, depending upon the QC type in question.
  - 20.1.1. MS/MSD/PDS: Acceptability of the MS/MSD/PDS recoveries is subject to the matrix and any anomalies associated with the subject batch. Failure of recoveries of an MS/MSD/PDS data set does not constitute an automatic reanalysis of the batch samples. Rather, it is acceptable to defer to the LCS/LCSD recoveries, to determine acceptance of the sample results.
  - 20.1.2. LCS/LCSD: Because they denote whether the analytical system is operating within control, it is imperative that the LCS recoveries obtained are within acceptance criteria. If the recoveries fail for a given reported element, the technical director confirms the unacceptable result.
    - 20.1.2.1. If the LCS results are verified as acceptable, no corrective action is required.
    - 20.1.2.2. If the LCS result is verified as out-of-control, and the subject element is to be reported in samples within that analytical batch, the samples reported with that failed element must be re-analyzed with a valid LCS recovery for the element.
    - 20.1.2.3. If the LCS result is verified as out-of-control, and the subject element is NOT to be reported in the samples within that analytical batch, the samples are not subject to reanalysis. No corrective action is required for that batch.

## 21. WASTE MANAGEMENT

- 21.1. The proper disposal of analytical samples and laboratory wastes is not only good laboratory practice, but also regulated by a variety of local, state, and federal laws. In order to remain compliant with these laws, and at the same time keep sample disposal costs at a minimum, the samples and wastes are identified, segregated, and either returned to the client (preferable) or placed into the proper laboratory waste stream.
- 21.2. Unused or remaining soil or liquid samples and all other solid or liquid wastes resulting from our laboratory operations are considered hazardous for disposal purposes.
- 21.3. All laboratory personnel must be aware of the types of chemicals they are using and the appropriate procedures for their disposal.
- 21.4. Each specific laboratory area shall maintain clearly labeled waste containers for small quantity waste collection. These waste containers shall be used for temporary collection of residual sample from aliquotting procedures, contaminated consumables, sample extracts, purged aqueous samples, and other wastes that require disposal as hazardous waste.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 42 of 70

- 21.5. To ensure compliance with Federal RCRA regulations, the Hazardous Waste Coordinator collects and disposes of the hazardous waste at each satellite collection point no less than monthly.
- 21.6. In order to maintain accountability for all samples received by Eurofins Calscience, when a sample is used in its entirety for analysis, the empty container(s) are returned to Sample Control for placement in analytical storage.
- 21.7. Waste management procedures shall adhere to the current revision of SOP-T005, "Disposal of Laboratory Samples and Wastes."

## 22. REFERENCES

- 22.1. *Inductively Coupled Plasma-Atomic Emission Spectrometry*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 6010B, USEPA, Revision 2, December 1996.
- 22.2. *Inductively Coupled Plasma-Atomic Emission Spectrometry*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 6010C, USEPA, Revision 3, November 2000.
- 22.3. *Flame Atomic Absorption Spectrophotometry*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1A, Method 7000B, USEPA, Revision 2, February 2007.
- 22.4. *Quality Control*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter One, USEPA, Revision 1, July 1992.
- 22.5. *Choosing the Correct Procedure*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Two, USEPA, Revision 4, February 2007.
- 22.6. *Inorganic Analytes*, Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Volume 1, Chapter Three, USEPA, Revision 4, February 2007.

## 23. APPENDICES, TABLES, DIAGRAMS, FLOWCHARTS AND VALIDATION DATA

- 23.1. Appendix A: Recommended Wavelengths and Estimated Instrumental Detection Limits.
- 23.2. Appendix B: Potential Interferences (Example), Analyte Concentration Equivalents Arising from Interference at the 100-mg/L Level.
- 23.3. Appendix C: Standard Solution Preparation.
- 23.4. Appendix D: Sample Holding Times, Required Digestion Volumes and Recommended Collection Volumes for Metal Determinations in Aqueous and Solid Samples.
- 23.5. Appendix E: Standard Addition Plot (Example).
- 23.6. Appendix F: Additional Quality Control Criteria for Department of Defense Projects.
- 23.7. Appendix G: Control Limits for Department of Defense Projects.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 43 of 70

## 24. MODIFICATIONS

24.1. The following modifications from EPA Method 6010B Revision 2 are noted.

ECI SOP M601 Section	Reference Document EPA Method 6010B Section	Summary of Modification
12.4. and 12.8.	7.4	The control limits of replicate %RSD are modified from < 5% to ≤ 5%.
12.5. and 12.9.	8.6.1.3	The acceptance criteria of ICB and CCB are modified.
12.14.	8.5.2	The spike concentration of PDS is modified from 10–100 times the IDL to 10–100 times the RL.
14.2.	7.2.5	Procedure on the determination of detection limit was modified to conform to the requirements specified in 40 CFR Part 136 Appendix B and 2009 TNI Standard.

## 25. ► REVISION HISTORY

Revision	Description	Author(s)	Effective Date
6.1	Entire document: Update company name. Section 6: Update definitions. Sections 8 and 17: Add SDS. Sections 19 and 20: Update responsibilities.	L. Hunt	2015-03-23
6.2	Section 6: Refer to QSM for the list of definitions and glossaries. Sections 8 and 17: Remove the references to MSDS. Section 9.2.2: Update the instrument software versions. Section 10.2: Add commercially prepared custom references standards. Section 19.2: Delete the reference to QA Director. Appendix F: Revise the whole appendix to conform to the DoD QSM Version 4.2. Appendix G: Revise the whole appendix to conform to the DoD QSM Version 4.2.	K. Chang	2016-05-02

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 44 of 70

---

## Appendix A

### RECOMMENDED WAVELENGTHS AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Eurofins Calscience, Inc.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 45 of 70

## Appendix A

### Recommended Wavelengths and Estimated Instrumental Detection Limits (IDLs)

Element	Detection Wavelength <sup>a</sup> (nm)	Estimated IDL <sup>b</sup> (µg/L)
Aluminum (Al)	308.215	30
Antimony (Sb)	217.582	21
Arsenic (As)	193.696	35
Barium (Ba)	233.527	0.87
Beryllium (Be)	313.042	0.18
Boron (B)	249.677 × 2	3.8
Cadmium (Cd)	226.502	2.3
Calcium (Ca)	317.933	6.7
Chromium (Cr)	267.716	4.7
Cobalt (Co)	228.616	4.7
Copper (Cu)	324.752	3.6
Iron (Fe)	273.955	4.1
Lead (Pb)	220.353	28
Lithium (Li)	610.362	2.8
Magnesium (Mg)	279.077	20
Manganese (Mn)	257.610	0.93
Molybdenum (Mo)	202.031	5.3
Nickel (Ni)	231.604 × 2	10
Phosphorus (P)	213.617	51
Potassium (K)	766.490	See note <sup>c</sup>
Selenium (Se)	196.026	50
Silica (SiO <sub>2</sub> )	251.611	17
Silver (Ag)	328.068	4.7
Sodium (Na)	589.592	19
Strontium (Sr)	407.771	0.28
Thallium (Tl)	190.801	27
Tin (Sn)	189.927	17
Titanium (Ti)	336.121	5.0
Vanadium (V)	292.402	5.0
Zinc (Zn)	213.857 × 2	1.2

<sup>a</sup> The wavelengths listed (where ×2 indicates second order) are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted (e.g., in the case of an interference) if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference (see Section 7.1.). In time, other elements may be added as more information becomes available and as required.

<sup>b</sup> The estimated instrumental detection limits shown are provided as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

<sup>c</sup> Highly dependent on operating conditions and plasma position.



STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 46 of 70

## Appendix B

### POTENTIAL INTERFERENCES (EXAMPLE)

Eurofins Calscience, Inc.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 47 of 70

## Appendix B

### Potential Interferences (Example)

Analyte Concentration Equivalents Arising from Interference at the 100-mg/L Level <sup>c</sup>

Analyte	Wavelength (nm)	Interferant <sup>ab</sup>									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ti	V	
Aluminum	Al	308.215	--	0.01926	0.01760	0.00290	--	0.00296	--	0.00470	0.63900
Antimony	Sb	206.836	--	--	1.50400	--	--	0.00116	--	--	--
Antimony	Sb	217.582	--	--	0.00910	--	--	0.00116	--	--	0.14900
Arsenic	As	188.979	--	--	--	0.00290	--	--	0.00440	0.00150	0.00040
Arsenic	As	193.696	--	0.00035	0.04780	--	--	--	--	--	--
Barium	Ba	233.527	0.00009	--	--	--	0.00403	0.00025	--	--	--
Beryllium	Be	313.042	--	--	0.00025	--	--	--	--	--	0.00650
Cadmium	Cd	226.502	--	--	--	--	0.00096	--	--	0.00005	0.00020
Calcium	Ca	317.933	0.00262	--	0.03450	0.00790	0.01350	0.02850	0.01330	0.00760	--
Chromium	Cr	267.716	0.00096	0.00009	--	0.02110	0.00186	0.00090	0.03080	0.00085	--
Cobalt	Co	228.616	0.00023	0.00001	--	0.00050	0.00166	0.00006	0.00045	0.00015	0.00050
Copper	Cu	324.752	0.00325	0.00229	--	--	--	0.00225	0.02680	0.04170	--
Iron	Fe	273.955	0.00617	--	0.01020	0.00660	--	0.00378	0.01140	0.00460	0.21900
Lead	Pb	220.353	--	--	--	0.00710	--	--	0.01530	0.00050	--
Magnesium	Mg	279.077	0.00066	0.00067	--	--	0.00049	--	--	0.00075	--
Manganese	Mn	257.610	--	0.00018	--	--	--	0.00041	--	0.00182	--
Molybdenum	Mo	202.031	0.00142	--	0.00625	0.00045	--	--	--	--	--
Nickel	Ni	231.604	0.00023	0.00002	0.00100	0.00010	0.00034	--	--	0.03940	0.00190
Phosphorus	P	213.617	--	--	--	0.87900	--	--	--	0.00430	0.01840
Potassium	K	766.490	--	--	--	--	--	0.00780	--	--	--
Selenium	Se	196.026	0.00667	--	--	0.00440	--	--	0.05840	--	0.00450
Silver	Ag	328.068	--	--	0.00040	0.00650	--	--	0.00045	--	--
Sodium	Na	589.592	0.09157	0.00801	0.10960	0.00580	--	0.13380	--	0.08005	--
Strontium	Sr	407.771	--	0.00258	0.00065	--	--	--	--	--	--
Thallium	Tl	190.801	0.00082	0.00378	0.04500	0.00445	0.00024	--	--	--	0.05500
Tin	Sn	189.927	0.00032	0.00301	--	0.00045	0.00059	--	0.00085	--	0.00165
Titanium	Ti	336.121	--	--	--	0.00015	0.00003	--	0.00010	0.00010	--
Vanadium	V	292.402	0.00011	--	--	--	--	0.00023	--	0.00025	--
Zinc	Zn	213.857	0.00041	0.00027	0.00285	0.05220	0.01660	0.00033	--	0.00100	--

<sup>a</sup> Dashes indicate that no interference was observed even when interferants were introduced at the following levels:

Al - 200 mg/L	Mg - 200 mg/L
Ca - 200 mg/L	Mn - 200 mg/L
Cr - 1000 mg/L	Ti - 1000 mg/L
Cu - 1000 mg/L	V - 1000 mg/L
Fe - 200 mg/L	

<sup>b</sup> The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferant figure.

<sup>c</sup> Interferences will be affected by background choice and other interferences may be present.

STANDARD OPERATING PROCEDURE  
Title: EPA 8010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 48 of 70

## Appendix C

### STANDARD SOLUTION PREPARATION

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STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 49 of 70

### Appendix C Initial Calibration Standard Solution Preparation

Initial Calibration Standard						
Element		Initial Conc (ppm)	Initial Vol (mL)	Final Conc (ppm)	Final Vol (mL)	Solvent Acid Conc <sup>a</sup>
Aluminum	Al	200 + 2000	60 + 7.5	27	1000	5% (v/v) HCl + 6% (v/v) HNO <sub>3</sub>
Antimony	Sb	200 + 1000	15 + 6	9	1000	
Arsenic	As	500	15	7.5	1000	
Barium	Ba	2000	7.5	15	1000	
Beryllium	Be	50 + 50	15 + 7.5	1.125	1000	
Boron	B	100 + 1000	15 + 6	7.5	1000	
Cadmium	Cd	100	15	1.5	1000	
Calcium	Ca	1000	60	60	1000	
Chromium	Cr	20	60	1.2	1000	
Cobalt	Co	500	7.5	3.75	1000	
Copper	Cu	250	7.5	1.875	1000	
Iron	Fe	1000	7.5	7.5	1000	
Lead	Pb	500	15	7.5	1000	
Magnesium	Mg	1000	15	15	1000	
Manganese	Mn	100	15	1.5	1000	
Molybdenum	Mo	200	6	1.2	1000	
Nickel	Ni	20	60	1.2	1000	
Phosphorus	P	10000	1.2	12	1000	
Potassium	K	400 + 10000	60 + 3	54	1000	
Selenium	Se	200	15	3	1000	
Silicon	Si	2000	6	12	1000	
Silver	Ag	50	15	0.75	1000	
Sodium	Na	200 + 10000	60 + 6	72	1000	
Strontium	Sr	10	60	0.6	1000	
Thallium	Tl	200	15	3	1000	
Tin	Sn	1000	6	6	1000	
Titanium	Ti	200	6	1.2	1000	
Vanadium	V	500	7.5	3.75	1000	
Zinc	Zn	100 + 10000	15 + 0.35	5	1000	
Bismuth <sup>b</sup>	Bi	1000	0.2	2	100	5% (v/v) HCl + 5% (v/v) HNO <sub>3</sub>
Lithium <sup>b</sup>	Li	1000	0.2	2	100	
Sulfur <sup>b</sup>	S	1000	0.2	2	100	

<sup>a</sup> HCl and HNO<sub>3</sub> are concentrated trace metals grade acids.

<sup>b</sup> Bi, Li, and S standards are prepared separately.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 50 of 70

### Appendix C

#### Initial Calibration Verification (ICV) Standard Solution Preparation

Initial Calibration Verification (ICV) Standard						
Element		Initial Conc (ppm)	Initial Vol (mL)	Final Conc (ppm)	Final Vol (mL)	Solvent Acid Conc <sup>a</sup>
Aluminum	Al	200	4	4	200	5% (v/v) HCl + 6% (v/v) HNO <sub>3</sub>
Antimony	Sb	200	2	2	200	
Arsenic	As	500	2	5	200	
Barium	Ba	100	2	1	200	
Beryllium	Be	50	2	0.5	200	
Boron	B	500	1	2.5	200	
Cadmium	Cd	150	2	1.5	200	
Calcium	Ca	1000	4	20	200	
Chromium	Cr	20	4	0.4	200	
Cobalt	Co	100	2	1	200	
Copper	Cu	100	2	1	200	
Iron	Fe	10000	2	100	200	
Lead	Pb	500	2	5	200	
Magnesium	Mg	1000	2	10	200	
Manganese	Mn	100	2	1	200	
Molybdenum	Mo	100 + 300	2 + 1	2.5	200	
Nickel	Ni	20	4	0.4	200	
Phosphorus	P	1000	1	5	200	
Potassium	K	400	4	8	200	
Selenium	Se	200	2	2	200	
Silicon	Si	230	1	1.15	200	
Silver	Ag	50	2	0.5	200	
Sodium	Na	200 + 10000	4 + 1	54	200	
Strontium	Sr	10	4	0.2	200	
Thallium	Tl	200	2	2	200	
Tin	Sn	10000	0.05	2.5	200	
Titanium	Ti	1000	1	5	200	
Vanadium	V	100	2	1	200	
Zinc	Zn	150	2	1.5	200	
Bismuth <sup>b</sup>	Bi	1000	0.1	1	100	5% (v/v) HCl + 5% (v/v) HNO <sub>3</sub>
Lithium <sup>b</sup>	Li	1000	0.1	1	100	
Sulfur <sup>b</sup>	S	1000	0.1	1	100	

<sup>a</sup> HCl and HNO<sub>3</sub> are concentrated trace metals grade acids.

<sup>b</sup> Bi, Li, and S standards are prepared separately.

STANDARD OPERATING PROCEDURE  
Title: EPA 8010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 51 of 70

### Appendix C

#### Low-Level Initial Calibration Verification (LLICV) Standard Solution Preparation

Low-Level Initial Calibration Verification (LLICV) Standard						
Element		Initial Conc (ppm)	Initial Vol (mL)	Final Conc (ppm)	Final Vol (mL)	Solvent Acid Conc <sup>a</sup>
Aluminum	Al	10000	0.5	5	1000	10% (v/v) HNO <sub>3</sub>
Antimony	Sb	10000	0.15	1.5	1000	
Arsenic	As	10000	0.1	1	1000	
Barium	Ba	10000	0.1	1	1000	
Beryllium	Be	10000	0.1	1	1000	
Boron	B	10000	0.2	2	1000	
Cadmium	Cd	10000	0.1	1	1000	
Calcium	Ca	10000	1	10	1000	
Chromium	Cr	10000	0.1	1	1000	
Cobalt	Co	10000	0.1	1	1000	
Copper	Cu	10000	0.1	1	1000	
Iron	Fe	10000	1	10	1000	
Lead	Pb	5000 <sup>b</sup>	0.2	1	1000	
Magnesium	Mg	10000	1	10	1000	
Manganese	Mn	10000	0.05	0.5	1000	
Molybdenum	Mo	10000	0.1	1	1000	
Nickel	Ni	10000	0.1	1	1000	
Phosphorus	P	10000	1	10	1000	
Potassium	K	10000	5	50	1000	
Selenium	Se	10000	0.15	1.5	1000	
Silicon	Si	10000	0.5	5	1000	
Silver	Ag	10000	0.05	0.5	1000	
Sodium	Na	10000	5	50	1000	
Strontium	Sr	10000	0.3	3	1000	
Thallium	Tl	10000	0.15	1.5	1000	
Tin	Sn	10000	0.5	5	1000	
Titanium	Ti	10000	0.3	3	1000	
Vanadium	V	10000	0.1	1	1000	
Zinc	Zn	10000	0.1	1	1000	
Bismuth <sup>b</sup>	Bi	1000	1	10	100	5% (v/v) HNO <sub>3</sub>
Lithium <sup>b</sup>	Li	1000	0.5	5	100	
Sulfur <sup>b</sup>	S	1000	1	10	100	
Lead	Pb	10000	100	5000	200	H <sub>2</sub> O

<sup>a</sup> HNO<sub>3</sub> is concentrated trace metals grade acid.

<sup>b</sup> Bi, Li, and S standards are prepared separately; 5000-ppm Pb standard is prepared separately.

STANDARD OPERATING PROCEDURE  
Title: EPA 8010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 52 of 70

### Appendix C

#### Continuing Calibration Verification (CCV) Standard Solution Preparation

Continuing Calibration Verification (CCV) Standard						
Element		Initial Conc (ppm)	Initial Vol (mL)	Final Conc (ppm)	Final Vol (mL)	Solvent Acid Conc <sup>a</sup>
Aluminum	Al	27	50	13.5	100	5% (v/v) HCl + 6% (v/v) HNO <sub>3</sub>
Antimony	Sb	9	50	4.5	100	
Arsenic	As	7.5	50	3.75	100	
Barium	Ba	15	50	7.5	100	
Beryllium	Be	1.125	50	0.5625	100	
Boron	B	7.5	50	3.75	100	
Cadmium	Cd	1.5	50	0.75	100	
Calcium	Ca	60	50	30	100	
Chromium	Cr	1.2	50	0.6	100	
Cobalt	Co	3.75	50	1.875	100	
Copper	Cu	1.875	50	0.9375	100	
Iron	Fe	7.5	50	3.75	100	
Lead	Pb	7.5	50	3.75	100	
Magnesium	Mg	15	50	7.5	100	
Manganese	Mn	1.5	50	0.75	100	
Molybdenum	Mo	1.2	50	0.6	100	
Nickel	Ni	1.2	50	0.6	100	
Phosphorus	P	12	50	6	100	
Potassium	K	54	50	27	100	
Selenium	Se	3	50	1.5	100	
Silicon	Si	12	50	6	100	
Silver	Ag	0.75	50	0.375	100	
Sodium	Na	72	50	36	100	
Strontium	Sr	0.6	50	0.3	100	
Thallium	Tl	3	50	1.5	100	
Tin	Sn	6	50	3	100	
Titanium	Ti	1.2	50	0.6	100	
Vanadium	V	3.75	50	1.875	100	
Zinc	Zn	5	50	2.5	100	
Bismuth <sup>b</sup>	Bi	2	50	1	100	5% (v/v) HCl + 5% (v/v) HNO <sub>3</sub>
Lithium <sup>b</sup>	Li	2	50	1	100	
Sulfur <sup>b</sup>	S	2	50	1	100	

<sup>a</sup> HCl and HNO<sub>3</sub> are concentrated trace metals grade acids.

<sup>b</sup> Bi, Li, and S standards are prepared separately.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 53 of 70

### Appendix C

#### Interference Check Standard AB (ICS-AB) Solution Preparation

Interference Check Standard AB (ICS AB)						
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume <sup>a</sup> (mL)	Solvent Acid Conc <sup>a</sup>
Aluminum	Al	1200	10	24	500	5% (v/v) HCl + 6% (v/v) HNO <sub>3</sub>
Antimony	Sb	500	1	1	500	
Arsenic	As	1000	0.5	1	500	
Barium	Ba	300	0.5	0.3	500	
Beryllium	Be	100	0.5	0.1	500	
Boron	B	500	0.5	0.5	500	
Cadmium	Cd	300	0.5	0.3	500	
Calcium	Ca	6000	10	120	500	
Chromium	Cr	300	0.5	0.3	500	
Cobalt	Co	300	0.5	0.3	500	
Copper	Cu	300	0.5	0.3	500	
Iron	Fe	5000	10	100	500	
Lead	Pb	1000	0.5	1	500	
Magnesium	Mg	3000	10	60	500	
Manganese	Mn	200	0.5	0.2	500	
Molybdenum	Mo	300	0.5	0.3	500	
Nickel	Ni	300	0.5	0.3	500	
Phosphorus	P					
Potassium	K	20000	0.5	20	500	
Selenium	Se	500	0.5	0.5	500	
Silicon	Si	200	0.5	0.2	500	
Silver	Ag	300	0.5	0.3	500	
Sodium	Na	1000	10	20	500	
Strontium	Sr					
Thallium	Tl	1000	0.5	1	500	
Tin	Sn					
Titanium	Ti	1000	0.5	1	500	
Vanadium	V	300	0.5	0.3	500	
Zinc	Zn	300	0.5	0.3	500	
Bismuth	Bi					
Lithium	Li					
Sulfur	S					

<sup>a</sup> HCl and HNO<sub>3</sub> are concentrated trace metals grade acids.



STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 54 of 70

### Appendix C

#### Interference Check Standard A (ICS-A) Solution Preparation

Interference Check Standard A (ICS A)						
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume <sup>a</sup> (mL)	Solvent Acid Conc <sup>a</sup>
Aluminum	Al	1200	10	24	500	5% (v/v) HCl + 6% (v/v) HNO <sub>3</sub>
Calcium	Ca	6000	10	120	500	
Iron	Fe	5000	10	100	500	
Magnesium	Mg	3000	10	60	500	
Sodium	Na	1000	10	20	500	

<sup>a</sup> HCl and HNO<sub>3</sub> are concentrated trace metals grade acids.

#### Internal Standard Solution Preparation

Internal Standard						
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume <sup>a</sup> (mL)	Solvent Acid Conc <sup>a</sup>
Holmium	Ho	10000	1	5	2000	5% (v/v) HCl + 6% (v/v) HNO <sub>3</sub>
Terbium	Tb	10000	1	5	2000	
Yttrium	Y	10000	1	5	2000	

<sup>a</sup> HCl and HNO<sub>3</sub> are concentrated trace metals grade acids.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 55 of 70

### Appendix C Spike Standard Solution Preparation

Spike Standards 1 & 2						
Element		Initial Conc. (ppm)	Initial Volume (mL)	Final Conc. (ppm)	Final Volume <sup>a</sup> (mL)	Solvent Acid Conc <sup>a</sup>
Aluminum	Al	10000	10	100	1000	10% (v/v) HNO <sub>3</sub>
Antimony	Sb	10000	10	100	1000	
Arsenic	As	10000	10	100	1000	
Barium	Ba	10000	10	100	1000	
Beryllium	Be	10000	10	100	1000	
Boron	B	10000	10	100	1000	
Cadmium	Cd	10000	10	100	1000	
Caicium	Ca	10000	10	100	1000	
Chromium	Cr	10000	10	100	1000	
Cobalt	Co	10000	10	100	1000	
Copper	Cu	10000	10	100	1000	
Iron	Fe	10000	10	100	1000	
Lead	Pb	5000 <sup>b</sup>	20	100	1000	
Magnesium	Mg	10000	10	100	1000	
Manganese	Mn	10000	10	100	1000	
Molybdenum	Mo	10000	10	100	1000	
Nickel	Ni	10000	10	100	1000	
Phosphorus	P	10000	10	100	1000	
Potassium	K	10000	100	1000	1000	
Selenium	Se	10000	10	100	1000	
Silicon	Si	10000	10	100	1000	
Silver	Ag	10000	5	50	1000	
Sodium	Na	10000	100	1000	1000	
Strontium	Sr	10000	10	100	1000	
Thallium	Tl	10000	10	100	1000	
Tin	Sn	10000	10	100	1000	
Titanium	Ti	10000	10	100	1000	
Vanadium	V	10000	10	100	1000	
Zinc	Zn	10000	10	100	1000	
Bismuth <sup>b</sup>	Bi	1000	20	200	100	5% (v/v) HNO <sub>3</sub>
Lithium <sup>b</sup>	Li	1000	20	200	100	
Sulfur <sup>b</sup>	S	1000	20	200	100	
Lead	Pb	10000	100	5000	200	H <sub>2</sub> O

<sup>a</sup> HNO<sub>3</sub> is concentrated trace metals grade acid.

<sup>b</sup> Bi, Li, and S standards are prepared separately; 5000-ppm Pb standard is prepared separately.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 56 of 70

#### Appendix D

#### SAMPLE HOLDING TIMES, REQUIRED DIGESTION VOLUMES AND RECOMMENDED COLLECTION VOLUMES FOR METAL DETERMINATIONS IN AQUEOUS AND SOLID SAMPLES

Eurofins Calscience, Inc.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 57 of 70

## Appendix D

### Sample Holding Times, Required Digestion Volumes and Recommended Collection Volumes for Metal Determinations in Aqueous and Solid Samples

Measurement	Digestion Volume (mL) <sup>a, c</sup>	Collection Volume (mL) <sup>a, c</sup>	Treatment/Preservative Holding Time <sup>b</sup>
<b><u>Inorganic Analytes</u> (except hexavalent chromium and mercury):</b>			
<b>Aqueous</b>			
Total	50	250	HNO <sub>3</sub> to pH < 2 6 months
Dissolved	50	250	Filter on site HNO <sub>3</sub> to pH < 2 6 months
Suspended	50	250	Filter on site 6 months
<b>Solid</b>			
Total	2 g	4 oz	6 months
<b><u>Hexavalent Chromium:</u></b>			
<b>Aqueous</b>			
	50	250	24 hours Store at 4 ± 2°C until analyzed
<b>Solid</b>			
	2.5 g	4 oz	1 month to extraction 4 days after extraction Store at 4 ± 2°C until analyzed
<b><u>Mercury:</u></b>			
<b>Aqueous</b>			
Total	50	250	HNO <sub>3</sub> to pH < 2 28 days
Dissolved	50	250	Filter HNO <sub>3</sub> to pH < 2 28 days
<b>Solid</b>			
Total	0.2 g	4 oz	28 days Store at 4 ± 2°C until analyzed

<sup>a</sup> Unless stated otherwise.

<sup>b</sup> Either glass or plastic containers may be used.

<sup>c</sup> Any sample volume reduction from the reference method's instructions must be made in the exact proportion as described in the method and representative sampling must be maintained.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 58 of 70

## Appendix E

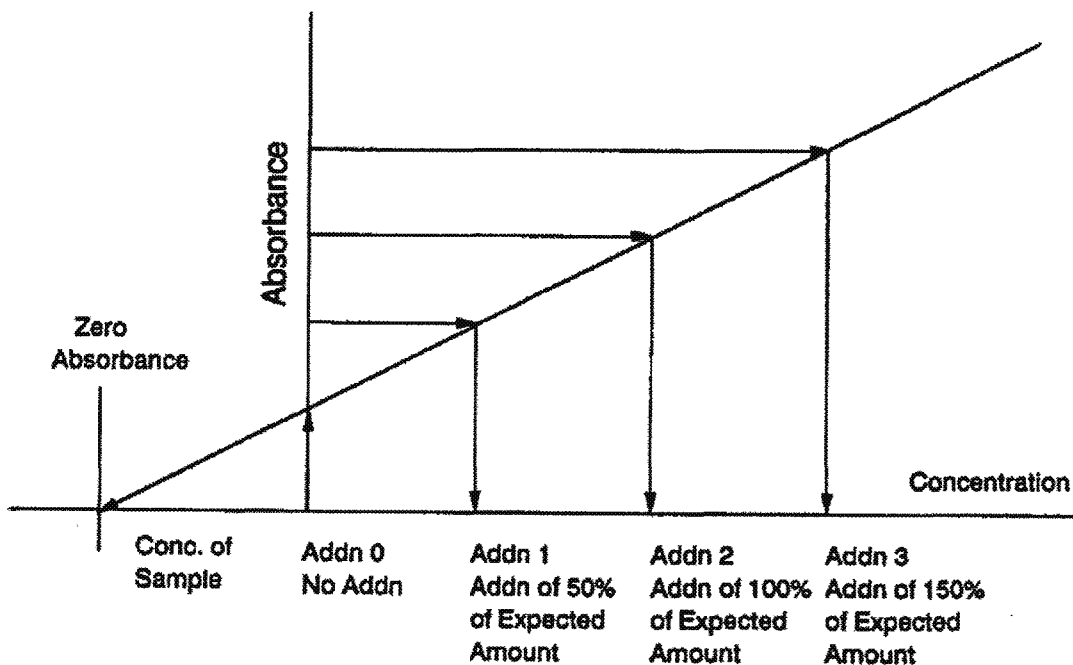
### STANDARD ADDITION PLOT (EXAMPLE)

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STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 59 of 70

**Appendix E**  
**Standard Addition Plot (Example)**



STANDARD OPERATING PROCEDURE  
Title: EPA 8010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 60 of 70

---

## Appendix F

### ADDITIONAL QUALITY CONTROL CRITERIA FOR DEPARTMENT OF DEFENSE PROJECTS

Eurofins Calscience, Inc.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 61 of 70

## 1. METHOD IDENTIFICATION

- 1.1. EPA Method 6010B, Inductively Coupled Plasma – Atomic Emission Spectrometry (ICP-AES) – Additional Quality Control Criteria for Department of Defense (DoD) Projects.

## 2. DETECTION / QUANTITATION LIMITS

- 2.1. The quantitation limit must be set within the calibration range.

## 3. SCOPE AND APPLICATION

- 3.1. The quality control criteria and procedure described herein either supersede or are in addition to the standard quality control criteria and procedure.

## 4. ►STANDARDS

- 4.1. Initial Calibration Verification (ICV)
  - 4.1.1. The concentration of the ICV standard shall be at or near the midpoint of the calibration range.
- 4.2. Low-Level Initial Calibration Verification (LLICV) Solution
  - 4.2.1. Prepare the LLICV working standard solutions by diluting the appropriate volumes of the stock standards and concentrated HNO<sub>3</sub> to the specified volumes with reagent water.
  - 4.2.2. Use the analyte and acid concentrations outlined in Appendix C as guidance to prepare the LLICV working standard solutions.
  - 4.2.3. Dilute the appropriate volumes of the LLICV working standard solution with reagent water for low-level initial calibration verification.
    - 4.2.3.1. Each target analyte in the LLICV solution is at a concentration expected to be the LOQ.
  - 4.2.4. Prepare the LLICV solution fresh daily.
  - 4.2.5. The LLICV solution is of a source same as that used for the initial one-point calibration.
  - 4.2.6. The concentration of the LLICV standard shall be less than or equal to the LOQ.
- 4.3. Continuing Calibration Verification (CCV)
  - 4.3.1. The concentration of the CCV standard shall be **between** the low calibration standard and the midpoint of the calibration range.
- 4.4. The use of a standard from a second lot **as a second source standard is acceptable when only one manufacturer of the standard exists. "Manufacturer" refers to the producer of the standard, not the vendor.**



STANDARD OPERATING PROCEDURE  
Title: EPA 8010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 62 of 70

## 5. ► QUALITY CONTROL

### 5.1. Limit of Detection (LOD)

- 5.1.1. Detection limit (DL) determination shall be performed for each analyte at the initial test method setup, following a change in the test method that affects how the test is performed, and following a change in instrumentation that affects the sensitivity of the analysis thereafter.
- 5.1.2. LOD verification must be performed immediately following each DL determination and quarterly thereafter.
  - 5.1.2.1. LOD verification sample shall be prepared by spiking a quality system matrix at **approximately 2 to 3 times the DL (for a single-analyte standard) or greater than 1 to 4 times the DL (for a multi-analyte standard)**.
  - 5.1.2.2. LOD verification is deemed valid if the apparent signal-to-noise (S/N) ratio of each analyte is at least 3 and the results must meet all method requirements for analyte identification.
    - 5.1.2.2.1. For a data system that does not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least 3 standard deviations greater than the mean method blank concentrations.
  - 5.1.2.3. If these criteria are not met, perform either one of the following tasks.
    - 5.1.2.3.1. Repeat the DL determination and LOD verification **at a higher concentration**.
    - 5.1.2.3.2. Perform and pass 2 consecutive LOD verifications at a higher concentration. Set the LOD at the higher concentration.

### 5.2. Limit of Quantitation (LOQ)

- 5.2.1. LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
  - 5.2.1.1. The procedure for establishing the LOQ must empirically demonstrate precision and bias at the LOQ for each analyte.
  - 5.2.1.2. The LOQ and associated precision and bias must meet client requirements and must be reported. If the test method is modified, precision and bias at the new LOQ must be demonstrated and reported.
- 5.2.2. LOQ verification must be performed quarterly to verify precision and bias at the LOQ.
  - 5.2.2.1. LOQ verification sample shall be prepared by spiking a quality system matrix at approximately 1 to 2 times the claimed LOQ.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 63 of 70

5.2.2.2. LOQ verification is deemed valid if the recovery of each analyte is within the established test method acceptance criteria or client data objectives for accuracy.

5.3. Initial Calibration (IC)

5.3.1. The LOQ and the calibration standard establish the quantitation range which must lie within the linear dynamic range.

5.3.1.1. When sample results exceed the quantitation range, dilute and re-analyze the sample (when sufficient sample volume and holding time permit) to bring results within the quantitation range. Results outside the quantitation range shall be reported as estimated values and qualified using appropriate data qualifiers that are explained in the case narrative.

5.4. Low-Level Initial Calibration Verification (LLICV)

5.4.1. Immediately following the analysis of an ICV standard, an LLICV standard must be analyzed prior to sample analysis.

5.4.2. The **accuracy of measurements at or near the RL is confirmed if** the %D for each analyte is  $\leq 20\%$ .

5.4.3. If these criteria are not met, **no sample analysis shall begin. Effect corrective action and re-analyze the LLICV.**

5.5. Initial Calibration Blank (ICB)

5.5.1. Immediately following the analysis of an LLICV standard, an ICB must be analyzed prior to sample analysis.

5.5.2. The instrument operating condition is deemed satisfactory for sample analysis to begin if no analytes are detected at a concentration  $> \text{LOD}$ .

5.5.3. If these criteria are not met, no sample analysis shall begin. Determine the source of contamination. Re-prepare and re-analyze the ICB.

5.6. Daily Spectral Interference Check (ICS-AB and ICS-A)

5.6.1. Following the establishment of a valid initial calibration, an ICS-AB and ICS-A solutions must be analyzed daily prior to sample analysis.

5.6.2. The ICS-AB is deemed acceptable if the %D for each analyte is  $\leq 20\%$ .

5.6.3. The ICS-A is deemed acceptable if the absolute value of the concentration for each non-spiked analyte is  $< \text{LOD}$  (unless it is a verified trace impurity from one of the spiked analytes).

5.6.4. If these criteria are not met, no sample analysis shall begin. Determine the source of problem, effect corrective action, and re-analyze the ICS-AB and/or ICS-A.

5.7. Continuing Calibration Verification (CCV)

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 64 of 70

- 5.7.1. Following the establishment of a valid initial calibration, a CCV standard must be analyzed daily after every batch of 10 field samples or portion thereof within a 24-hour shift, and at the end of sequence.
- 5.7.2. The initial calibration is deemed valid if the %D for each analyte is  $\leq 10\%$ .
- 5.7.3. If these criteria are not met, the initial calibration is deemed unacceptable for sample analysis to resume. **Effect corrective action** and re-analyze the CCV.
  - 5.7.3.1. If the **CCV remains unacceptable**, recalibrate and re-analyze all samples since the last acceptable CCV.
- 5.8. Continuing Calibration Blank (CCB)
  - 5.8.1. Immediately following the analysis of a CCV standard, a CCB must be analyzed prior to sample analysis.
  - 5.8.2. The instrument operating condition is deemed satisfactory for sample analysis to resume if no analytes are detected at a concentration  $> \text{LOD}$ .
  - 5.8.3. If these criteria are not met, no sample analysis shall resume. Determine the source of contamination. Re-prepare and re-analyze the CCB. Re-analyze all samples since the last acceptable CCB.
    - 5.8.3.1. **The results shall be reported with the appropriate data qualifier (B-flag) for the specific analyte(s) in all samples associated with the failed CCB.**
- 5.9. Event Based Quality Control (MBs and LCS/LCSDs)
  - 5.9.1. Method Blanks (MBs)
    - 5.9.1.1. The MB shall be considered to be contaminated if one of the following conditions is met.
      - 5.9.1.1.1. The concentration of any target analyte in the MB exceeds  $1/2$  the **RL**, and is greater than  $1/10$  the amount measured in any associated sample or  $1/10$  the regulatory limit (whichever is greater).
      - 5.9.1.1.2. The concentration of any common laboratory contaminant in the MB exceeds **RL**, and is greater than  $1/10$  the amount measured in any sample or  $1/10$  the regulatory limit (whichever is greater).
      - 5.9.1.1.3. **The MB result otherwise affects the sample results as per the test method requirements or the project specific data quality objectives (DQOs).**
    - 5.9.1.2. If the MB is contaminated, re-process the affected samples associated with the failed MB in a subsequent preparation batch, except when the sample results are below the LOD.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 65 of 70

5.9.1.2.1. If insufficient sample volume remains for re-processing, the results shall be reported with the appropriate data qualifier (B-flag) for the specific analyte(s) in all samples associated with the failed MB.

5.9.2. Laboratory Control Samples (LCS/LCSDs)

5.9.2.1. The lower and upper acceptance limits for %REC of each LCS/LCSD element in aqueous and solid matrices are listed in Appendix G.

5.9.2.2. All reported analytes must be spiked. The concentration of each spike analyte shall be at or below the midpoint of the calibration if project specific concentration is not specified.

5.9.2.3. Project-specific control limits shall be applied. If project-specific control limits are unavailable, DoD generated control limits shall be applied. If DoD generated control limits are unavailable, laboratory's in-house control limits shall be applied.

5.9.2.3.1. Laboratory's in-house control limits may not be greater than  $\pm 3S$  of the average recovery if the control limits are statistically-derived based on historical data with at least 30 data points generated under the same analytical process.

5.9.2.4. All project-specific analytes of concern must be within control limits. No marginal exceedance is allowed for any project-specific analyte of concern. If a project-specific analyte of concern exceeds its control limit, determine the cause of the problem and effect corrective action.

5.10. Matrix Based Quality Control (MS/MSDs)

5.10.1. Matrix Spikes (MS/MSDs)

5.10.1.1. The lower and upper acceptance limits for %REC of each MS/MSD element in aqueous and solid matrices are listed in Appendix G. The RPD is  $\leq 20\%$  (**between MS and MSD**).

5.10.1.2. All reported analytes must be spiked. The sample selected for spiking must be one of the samples collected for the specific DoD project.

5.10.1.3. Project-specific control limits shall be applied. If project-specific control limits are unavailable, DoD generated control limits shall be applied. If DoD generated control limits are unavailable, laboratory's in-house control limits shall be applied.

5.10.1.3.1. Laboratory's in-house control limits may not be greater than  $\pm 3S$  of the average recovery if the control limits are statistically-derived based on

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 66 of 70

historical data with at least 30 data points generated under the same analytical process.

5.11. Dilution Test

- 5.11.1. If **an analyte concentration** is  $> 50 \times \text{LOQ}$ , prepare a dilution test sample per preparation batch per matrix with a 1:5 dilution.
- 5.11.2. The analyte concentration in the dilution test sample must be within  $\pm 10\%$  of the original determination.
- 5.11.3. If this criterion is not met, **perform post digestion spike addition**.

5.12. Post Digestion Spike (PDS) Addition

- 5.12.1. If **the dilution test fails or an analyte concentration in all samples** is  $< 50 \times \text{LOD}$ , prepare a PDS sample per preparation batch per matrix.
- 5.12.2. A PDS sample is prepared by adding the spike standard to a portion of a digested sample, or its dilution. The spike addition should produce a concentration of 10–100 times the LOQ.
- 5.12.3. The acceptance criteria for PDS elements are as follows:
  - 5.12.3.1. The lower and upper acceptance limits for %REC of each PDS element are **75%** and **125%**, respectively.
  - 5.12.3.2. If these criteria are not met, **perform MSA on all samples in the same preparation batch**.

6. ►PROCEDURE

- 6.1. Following the establishment of a valid initial calibration, an LLICV standard must be analyzed daily immediately following ICV.
  - 6.1.1. If LLICV fails, effect corrective action prior to analyzing any samples.
- 6.2. Blank, standard, and sample vessels are loaded in the following or other logical order:
  - 1) Calibration Blank (CB)
  - 2) Initial Calibration Standard
  - 3) Initial Calibration Verification (ICV)
  - 4) Low-Level Initial Calibration Verification (LLICV)
  - 5) Initial Calibration Blank (ICB)
  - 6) Interference Check Solution AB (ICS-AB)
  - 7) Interference Check Solution A (ICS-A)
  - 8) Continuing Calibration Verification (CCV)
  - 9) Continuing Calibration Blank (CCB)
  - 10) Method Blank (MB)
  - 11) Laboratory Control Samples (LCS)
  - 12) Laboratory Control Sample Duplicates (LCSD)
  - 13) Samples (up to 10 per batch, including QC check samples and MBs)
  - 14) Matrix Spike (MS)

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 67 of 70

- 15) Matrix Spike Duplicate (MSD)
- 16) Dilution Test Sample
- 17) Post Digestion Spike (PDS)
- 18) Ending CCV
- 19) Ending CCB

- 6.2.1. Item 4: An LLICV is a standard used to confirm the accuracy of measurement at or near the **RL**. An acceptable LLICV is required daily immediately following ICV.
- 6.2.2. Item 5: The ICB is an aliquot of acidified reagent water used to monitor contamination. An acceptable ICB is required immediately following LLICV.
- 6.2.3. Item 16: The dilution test sample is prepared from the five-fold dilution of a sample **with an analyte concentration greater than 50 × LOQ** post digestion. The sample is diluted to one-fifth of the original concentration post digestion to confirm that no interference is observed in the original sample.
  - 6.2.3.1. One dilution test sample is required for every batch of 20 samples per matrix or portion thereof processed concurrently.
- 6.2.4. Item 17: The PDS is the same sample matrix from which the MS/MSD samples were prepared or from another sample in the same preparation batch, and is spiked with known concentrations of specific target analytes post digestion. The sample which will be spiked for the PDS is processed concurrently with the associated samples. In the processing of the PDS, reagents and procedures identical to those for actual samples are used.

## 7. REFERENCES

- 7.1. ►Department of Defense Quality Systems Manuals for Environmental Laboratories, Version **4.2, October 2010**.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 68 of 70

---

## Appendix G

### CONTROL LIMITS FOR DEPARTMENT OF DEFENSE PROJECTS

Eurofins Calscience, Inc.

STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.

Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 69 of 70

## Appendix G

### DoD Control Limits of LCS/LCSD/MS/MSD Elements in Aqueous Matrix

Analyte		Control Limit		ME Limit	
		Lower	Upper	Lower	Upper
Aluminum	Al	80	120	80	120
Antimony	Sn	80	120	80	120
Arsenic	As	80	120	80	120
Barium	Ba	80	120	80	120
Beryllium	Be	80	120	80	120
Cadmium	Cd	80	120	80	120
Calcium	Ca	80	120	80	120
Chromium	Cr	80	120	80	120
Cobalt	Co	80	120	80	120
Copper	Cu	80	120	80	120
Iron	Fe	80	120	80	120
Lead	Pb	80	120	80	120
Magnesium	Mg	80	120	80	120
Manganese	Mn	80	120	80	120
Molybdenum	Mo	80	120	75	120
Nickel	Ni	80	120	80	120
Potassium	K	80	120	80	120
Selenium	Se	80	120	75	120
Silver	Ag	80	120	75	120
Sodium	Na	80	120	80	120
Thallium	Tl	80	120	80	120
Vanadium	V	80	120	80	120
Zinc	Zn	80	120	80	120

Note: ME limits are applicable to LCS/LCSD elements only.



STANDARD OPERATING PROCEDURE  
Title: EPA 6010B, INDUCTIVELY COUPLED PLASMA – ATOMIC EMISSION  
SPECTROMETRY (ICP-AES)  
Eurofins Calscience, Inc.


Document No.: SOP-M601  
Revision No.: 6.2  
Effective Date: 2016-05-02  
Page 70 of 70

## Appendix G

### DoD Control Limits of LCS/LCSD/MS/MSD Elements in Solid Matrix

Analyte		Control Limit		ME Limit	
		Lower	Upper	Lower	Upper
Aluminum	Al	80	120	75	120
Antimony	Sn	80	120	75	120
Arsenic	As	80	120	80	120
Barium	Ba	80	120	80	120
Beryllium	Be	80	120	80	120
Cadmium	Cd	80	120	80	120
Calcium	Ca	80	120	80	120
Chromium	Cr	80	120	80	120
Cobalt	Co	80	120	80	120
Copper	Cu	80	120	80	120
Iron	Fe	80	120	80	120
Lead	Pb	80	120	80	120
Magnesium	Mg	80	120	80	120
Manganese	Mn	80	120	80	120
Molybdenum	Mo	80	120	75	120
Nickel	Ni	80	120	80	120
Potassium	K	80	120	80	120
Selenium	Se	80	120	75	120
Silver	Ag	75	120	70	125
Sodium	Na	80	120	80	120
Thallium	Tl	80	120	80	120
Vanadium	V	80	120	80	120
Zinc	Zn	80	120	75	120

Note: ME limits are applicable to LCS/LCSD elements only.

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Title : **40 CFR, Part 50 Appendix J; PM-10 Gravimetric  
Measurement; and Total Suspended Particulates in Air (TSP)**

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*Changes in this revision are noted in bold, italicized typeface and are preceded by a "►" marker.*

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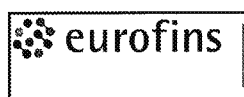
  
MANAGEMENT

10/16/2017  
DATE

  
QA DEPARTMENT

10-16-17  
DATE

Revision: 0.1	Effective Date: 10/16/17	Page 1 of 9
COMPANY CONFIDENTIAL		

	Calscience	Document Title: PM-10 Gravimetric Measurement; Total Suspended Particulates in Air (TSP)	Revision : 0.1  Supersedes : 0.0
---	------------	--	--

► **Reference:**

*Health, Safety, and Respiratory Protection Program Manual*, Eurofins Calscience, Inc., current version.

"Particulate Matter (PM-10)" *1995 National Air Quality: Status and Trends*. February 22, 2016. <https://www3.epa.gov/airtrends/aqtrnd95/pm10.html>

*Quality Systems Manual for Environmental Analytical Services*, Eurofins Calscience, Inc., current version.

"Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere" *Code of Federal Regulations*. Title 40 Chapter I Subchapter C Part 50 Appendix J. July 1, 2000

► **ASTM Standard, 2003, "Standard Test Method for the Determination of Total Suspended Particulate Matter in the Atmosphere (High-Volume Sampler Method)." ASTM International, West Conshohocken, PA, 2003, D4096-91, [www.astm.org](http://www.astm.org)**

**Cross Reference:**

Document	Document Title
T010	Employee Training
QSM	Eurofins Calscience Quality Systems Manual
	Health, Safety, and Respiratory Protection Program Manual


► **Scope:**

► This method is applicable to PM 10 filter samples **and Total Suspended Particulate (TSP) samples** field-tested by clients.

**Background Information:**

Particulate matter with a diameter of 10 µm has been proven to be harmful to human health, particularly to the lungs and respiratory system.

Revision: 0.1	Effective Date: 10/16/17	Page 2 of 9
COMPANY CONFIDENTIAL		

 eurofins	Calscience	Document Title:	Revision : 0.1
		PM-10 Gravimetric Measurement; Total Suspended Particulates in Air (TSP)	Supersedes : 0.0

### Basic Principles:

Particle size collection instruments use a specifically designed opening and only allow entry to air and particulate matter within a specific diameter range. Inside the instrument is a pre-weighed filter, which will retain any particulate matter with a diameter greater than 2.2  $\mu\text{m}$ . The client uses these instruments for a known amount of time and at a specific rate of air flow. After analysis, the filter is weighed and the mass of particulate matter on the filter is calculated. The flow rate and the length of time the instrument was run are used to calculate the volume of air that passed through the filter. Finally, the concentration of particulate matter is determined, using the mass on the filter and the volume of air.

### Reference Modifications:

The laboratory cannot reproduce testing conditions (collection instrument, analyst, etc.,) due to the nature of sample collection. Our quality control procedures are described in the Quality Control section of this SOP.

### Definitions:

**PM 10:** particulate matter with a diameter of 10 micrometers or less.


Definitions of other terms used in this SOP are found in the Quality Systems Manual, Appendix 1.

### ► Interferences:

Interferences occur through chemical and physical means. These interferences are listed below:

1. Volatile Particles will be lost if filters must be transported or stored. Filters shall be weighed as soon as possible to prevent this error.
2. Filters may retain extra sulfur dioxide or nitric acid (artifact sulfur or nitrate formation). This can be minimized by the use of specific filter types.
3. Particle deposits can be lost due to chemical reaction on the filter's surface or agitation. Filters shall always be stored in a desiccator, transported in their envelopes, and handled carefully.
4. **► If a high volume of particulates is deposited on the filter, the air flow rate may decrease, causing an artificially low result when reported in units of mass per volume of air. According to ASTM, any sample should be suspect if the final flow rate is  $< \frac{1}{2}$  the initial flow rate. This information may not be available to the analyst.**

Revision: 0.1	Effective Date: 10/16/17	Page 3 of 9
COMPANY CONFIDENTIAL		

 eurofins	Calscience	Document Title: PM-10 Gravimetric Measurement; Total Suspended Particulates in Air (TSP)	Revision : 0.1 Supersedes : 0.0
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### Safety Precautions and Waste Handling:

All laboratory waste is accumulated, managed, and disposed of in accordance with all federal, state, and local laws and regulations. See the *Health, Safety, and Respiratory Protection Program Manual* for general information regarding employee safety, waste management, and pollution prevention.

Normal laboratory safety practices must be followed when performing this method.

► ***If desired, method blank filters used in previous batches may be re-used and given to a client if the initial and final weights of the filter were within 5 mg.***

### Personnel Training and Qualifications:

All personnel performing this procedure must have documentation of reading, understanding, and agreeing to follow the current version of this SOP and a documented Demonstration of Capability.

Demonstration of Capability consists of four method blank samples that are carried through all steps of the analysis and meet the defined acceptance criteria. The method blanks must be within  $\pm 10\%$  of the original sample weight.

### ► Sample Collection, Preservation, and Handling:

PM10 filters will be stored in a desiccator under the conditions below until requested by the clients. When they return from the client, they will be stored in a desiccator in the same conditions in a sample envelope. At this point, filters may be folded in half to prevent particulates from adhering to the envelope. Care will be taken to prevent further bending and agitation of the sample.


► Stored filters will remain between 20 to 45% RH and 15-27°C. The humidity of the desiccators shall be monitored using a NIST traceable hygrometer, which is replaced yearly. Normal laboratory temperature is maintained within the temperature limits. If there is a catastrophic failure of the laboratory's environmental control system, the client shall be notified of any potential effects to samples.

The sample holding time is 72 hours from sample collection.

### Apparatus and Equipment:

1. Analytical Balance
2. ► ***Sample collection filters:***
  - a) ***PM-10:*** Whatman QM-A 8x10 Quartz Filters (US Ref.: WHA1851-8866), or equivalent.

Revision: 0.1	Effective Date: 10/16/17	Page 4 of 9
COMPANY CONFIDENTIAL		

	Calscience	Document Title: PM-10 Gravimetric Measurement; Total Suspended Particulates in Air (TSP)	Revision : 0.1  Supersedes : 0.0
---	------------	--	--

- b) **TSP: Tisch Environmental 8x10 Glass Fiber Filters (SKU: TE-G653), or equivalent.**
3. Desiccator with hygrometer, Fisher Scientific Traceable Digital meter, Cat. No. S04709, or equivalent.
4. 500 mL beakers
5. **Filter envelopes**

► **Procedure:**


1. Prior to Sample Collection

- a) ► Store pre-numbered filters in a desiccator and allow to acclimate for 24 hours. **Usually, all filters in a pack are placed in the desiccator when the package is opened.**
- b) Place a 500-mL glass beaker in the analytical balance and tare.
- c) Gently roll the filter and place in the beaker. Avoid folding or creasing the filter. Record the sample weight. Remove the filter, inspect for imperfections, and record relevant information in the PM 10 Initial Weight logbook.
- d) Distribute filter to the client in a sample envelope.

2. After Sample Collection

- a) Leave sample in envelope and place in a desiccator for 24 hours. Identify a method blank for the sample batch and place in the desiccator with the samples. Record date and time filter is placed in and removed from desiccator.
  - b) Weigh samples and QC
    - i) Place 500-mL glass beaker in the analytical balance and tare.
    - ii) Remove the filter from the envelope, gently roll, and place in the beaker.
    - iii) Pour particulate matter from the envelope into the beaker.
    - iv) Weigh sample and record mass in the logbook.
    - v) Remove sample. Wipe beaker with a Kim wipe and repeat for each sample.
- (1) If particulate matter was poured from the envelope, pour it back into the envelope and wash the beaker to prevent contamination of the samples.

Revision: 0.1	Effective Date: 10/16/17	Page 5 of 9
COMPANY CONFIDENTIAL		

	Calscience	Document Title: PM-10 Gravimetric Measurement; Total Suspended Particulates in Air (TSP)	Revision : 0.1  Supersedes : 0.0
---	------------	--	--

- vi) ► **NOTE: A second conditioning and weighing step may be performed upon client request. In this case, results will be reported according to client specifications.**

#### Calculations:

Some customers will provide data sheets with the filters that describe sample conditions (such as temperature, operation time, etc.). These sheets will hold some information used in the calculations below.

► **NOTE: Analysts should check initial and final flow rates. If both are provided on the data sheets and the final flow rate is  $< \frac{1}{2}$  the initial flow rate, a note must be made on the data report.**

$$\text{Net weight} = \text{Final Weight} - \text{Initial Weight}$$

#### Where:

Net Weight = Mass of PM10

Final Weight = Final mass of filter and sample

Initial Weight = Initial mass of filter only

$$A = v \times t$$

#### Where:

A = Volume of air

v = flow rate

t = time

$$C_{PM10} = \frac{\text{Net Weight}}{A}$$

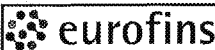
#### Where:

$C_{PM10}$  = Concentration of PM10 (Total Particulates)

Net weight = Mass of PM10

A = Volume of air

Revision: 0.1	Effective Date: 10/16/17	Page 6 of 9
COMPANY CONFIDENTIAL		

	Calscience	Document Title: PM-10 Gravimetric Measurement; Total Suspended Particulates in Air (TSP)	Revision : 0.1  Supersedes : 0.0
---	------------	--	--

$$v_{std} = v_a \times \left( \frac{P_{av}}{T_{av}} \right) \left( \frac{T_{std}}{P_{std}} \right)$$

**Where:**

$v_{std}$  = Flow rate at EPA reference conditions  $m^3/min$

$v_a$  = Flow rate at ambient conditions

$P_{av}$  = Average barometric pressure during the sampling period or average barometric pressure for the sampling site, kPa (or mm Hg)

$T_{av}$  = Average ambient temperature during the sampling period or seasonal average ambient temperature for the sampling site, K.

$T_{std}$  = Standard temperature (298 K)

$P_{std}$  = Standard pressure (101.3 kPa or 760 mm Hg)

**Statistical Information/Method Performance:**

The reporting limit is determined by the sensitivity of the analytical balance and is found in the relevant test code.

**Quality Assurance/Quality Control:**

► **For TSP, the minimum detection limit is 3 mg, as specified by the reference method.**

Analyze method blanks at a rate of 1 per batch of 20 samples. The method blank is a pre-weighed, unused filter that is stored in the desiccator with the received client samples. The final weight of the method blank must be within 5 **mg** of the initial weight. If the weight is above what is allowed, flag the associated samples.


► Analyze one replicate sample measurement at a rate of 1 per batch of 20 samples. A replicate measure involves returning the filter to the state it was received, taring the balance, and re-weighing the sample. This is meant to be an indicator of the amount of PM10 lost due to laboratory handling. **The RPD between replicate samples must be ≤10%.**

Any visible defects, punctures, or rips in the filter shall be noted in the logbook. In addition, any significant amount of particulate matter in the sample envelope shall be noted in the logbook.

Analyses requiring an LCS do not pertain to this method, because the laboratory cannot reproduce testing conditions (collection instrument, analyst, etc.). It is the responsibility

Revision: 0.1	Effective Date: 10/16/17	Page 7 of 9
COMPANY CONFIDENTIAL		




	Calscience	Document Title: PM-10 Gravimetric Measurement; Total Suspended Particulates in Air (TSP)	Revision : 0.1 Supersedes : 0.0
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of the client to monitor error from the collection instrument. ECI will weigh client-provided QC samples in the same manner as other client samples.

### Revision Log:

Revision: 0.1		Effective Date: This version
Section	Justification	Changes
		New

Revision: 0.1	Effective Date: 10/16/17	Page 8 of 9
COMPANY CONFIDENTIAL		

 eurofins	Calscience	Document Title: PM-10 Gravimetric Measurement; Total Suspended Particulates in Air (TSP)	Revision : 0.1 Supersedes : 0.0
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## Appendix I

### Quick Steps:

#### Before Client

1. Allow to acclimate in a desiccator for 24 hours.
2. Weigh filters by rolling and placing in a tared, 500-mL beaker. Record mass and filter number in logbook.

---

#### After Client

1. Allow samples to acclimate in a desiccator for 24 hours. Designate a method blank that will be analyzed with the sample batch.
2. Weigh samples and QC by removing from envelope, carefully rolling filters, and placing in a tared, 500-mL beaker.
3. Pour any particulate matter from the envelope into the beaker.
4. Weigh sample.
5. Wipe the beaker with a Kim wipe, repeat for all samples.
  - a) If particulates fell into the beaker, wash it to prevent contamination.
6. ► ***Check initial and final flow rates on client's data form. If both are provided on the data sheets and the final flow rate is < ½ the initial flow rate, a note must be made on the data report.***

Revision: 0.1	Effective Date: 10/16/17	Page 9 of 9
COMPANY CONFIDENTIAL		



Environment Testing  
TestAmerica

TestAmerica St. Louis

SOP No. ST-RC-0004, Rev. 28

Effective Date: 09/23/2019

Page No.: 1 of 17<sup>18</sup>

K2  
9/9/19

**Title: PREPARATION OF SOIL, SLUDGE, FILTER, BIOTA AND OIL & GREASE SAMPLES FOR RADIOCHEMICAL ANALYSES**

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## 1.0 SCOPE AND APPLICATION

- 1.1 This SOP has destructive procedures, which prepare a sample for radiometric measurement. This procedure is applicable to soils, sludge, filters, biota and oil/grease samples for many radiochemical analyses, including (but not limited to): isotopic americium, curium, neptunium, plutonium, thorium and uranium, Se-79, Fe-55, Pb-210, Ni-59/63, Ra-226, Ra-228, and Sr-90.
- 1.2 For samples requiring an acid leaching, the entire sample or a portion of the sample, may be leached and a leachate aliquot taken for all radiochemistry analyses. Method Blanks and QC samples are initiated after the leaching process.
- 1.3 The reporting limits and QC limits are maintained in the Laboratory Information Management System (LIMS).

## 2.0 SUMMARY OF METHOD

- 2.1 This SOP describes the method that is used to prepare a sample for analysis. After treatment with concentrated or dilute acids, sodium hydroxide fusion, and/or the muffle furnace, the sample extract or residue is transferred to the applicable procedure for further separation prior to analysis.

## 3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

## 4.0 INTERFERENCES

- 4.1 Inherent in this procedure is the assumption that the carrier and/or tracer solution mixes completely with the sample matrix. Incomplete mixing can cause anomalous chemical yield data.
- 4.2 Samples containing naturally high concentrations of the carriers and/or tracers (e.g. Ba, or U-232) can cause chemical yields in excess of 100% for some analyses.

## 5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
  - 5.2.1 None.
- 5.3 PRIMARY MATERIALS USED
  - 5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in

the SDS for each material before using it for the first time or when there are major changes to the SDS.

Compound	Hazards	Exposure Limits (1)	Signs of Exposure
Hydrofluoric Acid	Poison Corrosive	3 ppm (TWA)	Inhalation symptoms may include sore throat, coughing, labored breathing and lung congestion/inflammation. Skin contact may cause serious burns which are not immediately apparent or painful. Symptoms of eye contact include redness, pain, and blurred vision.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.
Hydrogen peroxide (30%)	Oxidizer Corrosive Fire (increases flammability of combustible, organic, and readily oxidizable materials)	1 ppm (TWA)	Irritation to respiratory tract and burning of mucous membrane of nose and throat. Pain, redness and blurred vision in eyes.
Hydrochloric Acid	Poison Corrosive	5 ppm (Ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
Sodium Hydroxide	Corrosive Poison	2 ppm (TWA)	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
1- Always add acid to water to prevent violent reactions.			
2- Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
Ceiling – At no time should this exposure limit be exceeded.			
STEL – Short term exposure limit			

## 6.0 EQUIPMENT AND SUPPLIES

- 6.1 Analytical balance.
- 6.2 Ashless Powder, cellulose filter, Whatman or equivalent.
- 6.3 Centrifuge tubes.
- 6.4 Disposable digestion tubes.
- 6.5 Air filters.
- 6.6 Griddle (Hot Plate), heating limit 450 °F.

- 6.7 Hot plates, stirring hotplates.
- 6.8 Mod Block digestion system.
- 6.9 Muffle furnace, programmable.
- 6.10 Teflon and Glass beakers, 50, 250, 2000 mL etc.
- 6.11 TEXPEN®, for high temperature marking of quartz crucibles.
- 6.12 Watch glasses, Teflon, glass and plastic.
- 6.13 Teflon stir bars.
- 6.14 Zirconium crucibles with lids.

## 7.0 REAGENTS AND STANDARDS

- 7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2. DI Water.
- 7.3. Aluminum nitrate solution (2M) (non-critical reagent)
  - 7.3.1 Dissolve and dilute 75 g aluminum nitrate into 100 mL with DI water.
- 7.4. Ammonium hydrogen phosphate solution (3.2M) (non-critical reagent)
  - 7.4.1 Dissolve 104 g of  $(\text{NH}_4)_2\text{HPO}_4$  in 200 mL of water, heat gently to dissolve, and dilute to 250 mL with water.
- 7.5. L (+) Ascorbic Acid, reagent powder
  - 7.5.1 L (+) Ascorbic Acid solution (~1.4M), 2.5 g dissolved in 10 mL of DI water (non-critical reagent).
- 7.6. Boric acid crystals,  $\text{H}_3\text{BO}_3$ , (non- critical Reagent).
  - 7.6.1 3M  $\text{HNO}_3$  and 0.25M Boric Acid
    - 7.6.1.1. In a 2.5L bottle, dissolve 31 g of Boric acid in approximately 1000 mL of DI water.
    - 7.6.1.2. Add approximately 380 mL of concentrated nitric acid.
    - 7.6.1.3. Dilute to 2000 mL using DI water.
- 7.7. Calcium nitrate solution (1.25 M) (non-critical reagent)
  - 7.7.1 Dissolve 51 g of  $\text{Ca}(\text{NO}_3)_2$  in 100 mL of water and dilute to 250 mL with water.
- 7.8. Eichrom Load solution, Nitric acid (3 M  $\text{HNO}_3$ ) in Aluminum nitrate (1 M  $\text{Al}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$ ) (non-critical reagent)
  - 7.8.1 Dissolve 350 g of Aluminum nitrate in approximately 700 mL of water.
  - 7.8.2 Add approximately 190 mL of concentrated nitric acid and dilute to 1000 mL with DI water.
- 7.9. Ferric nitrate nonahydrate crystals ( $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ).
  - 7.9.1 Ferric nitrate solution (50 mg/mL Fe), Non-critical reagent: Dissolve and dilute 36.2 g of  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  into 100 mL with DI water.

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- 7.9.2 Ferric nitrate solution (5 mg/mL Fe), Non-critical reagent: Dissolve and dilute 3.62 g of  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  into 100 mL with DI water. Alternatively, dilute 1 part ferric nitrate solution (50 mg/mL Fe) with 9 parts of DI water.
- 7.10. Hydrochloric acid (12M) – concentrated HCl (sp gr 1.19, 37.2%).
- 7.10.1 Hydrochloric acid (1.5M) – Non-critical reagent: Add 125 mL of concentrated HCl to 800 mL of DI water and dilute to 1 L. Mix well.
- 7.10.2 Hydrochloric acid (0.01M) – Non-critical reagent: Add 0.83 mL of concentrated HCl to 800 mL DI water and dilute to 1L. Mix well.
- 7.11. Hydrofluoric acid (48-52% HF) - concentrated
- 7.12. Lanthanum nitrate solution (10 mg/mL La), Non-Critical reagent: Dissolve and dilute 3.12 g of lanthanum nitrate hexahydrate into 100 mL with 1M  $\text{HNO}_3$ .
- 7.13. Nitric acid (16 M  $\text{HNO}_3$ ) -concentrated, sp. gr. 1.42, 70.4%.
- 7.13.1 Nitric Acid (8 M  $\text{HNO}_3$ ) – Non-Critical reagent: To a pre-marked 2.5L bottle containing 1000 mL DI water, add approximately 1000 mL concentrated  $\text{HNO}_3$ . Mix well.
- 7.13.2 Nitric Acid (7M  $\text{HNO}_3$ ) – Non-critical reagent: Add 443 mL of concentrated nitric acid to 500 mL DI water and dilute to 1L. Mix well.
- 7.13.3 Nitric Acid (4M  $\text{HNO}_3$ ) –Non-Critical reagent: to an appropriately sized bottle containing approximately 500 mL DI water, add approximately 250 mL concentrated  $\text{HNO}_3$  and dilute to 1L. Mix well.
- 7.13.4 Nitric Acid (3 M  $\text{HNO}_3$ ) – Non-Critical reagent: Add 375 mL of concentrated nitric acid to 1500 mL of DI water and dilute to 2 L. Mix well.
- 7.13.5 Nitric Acid (2 M  $\text{HNO}_3$ ) – Non-critical reagent: To a pre-marked 2.5L bottle containing 500 mL DI water, add 125 mL concentrated  $\text{HNO}_3$ , dilute to 1000 mL with DI water. Mix well.
- 7.13.6 Nitric Acid (1 M  $\text{HNO}_3$ ) – Non- critical reagent: To an appropriately sized bottle containing approximately 900 mL DI water, add approximately 62.5 mL concentrated  $\text{HNO}_3$  and dilute to 1 liter. Mix well.
- 7.14. Hydrogen Peroxide ( $\text{H}_2\text{O}_2$ ), 30%.
- 7.15. Sodium Carbonate ( $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ ), (1 M)
- 7.15.1 In a 1 L graduated cylinder dissolve 500 g of sodium carbonate in DI water. Dilute to a final volume of 1 L with DI water. Mix thoroughly and allow to settle overnight.
- 7.16. Sodium Hydroxide Pellets
- 7.17. Sodium Nitrite,  $\text{NaNO}_2$  reagent crystals
- 7.17.1 Sodium Nitrite solution (3.5 M) – Non-critical reagent: Dissolve and dilute 12.0 g of  $\text{NaNO}_2$  into 50 mL with DI water.
- 7.18. Titanium trichloride,  $\text{TiCl}_3$ , 20% solution, commercially available.
- 7.19. Americium 243 tracer, NIST traceable, approximately 10-20 dpm/mL.
- 7.20. Americium 241 spike, NIST traceable, approximately 10-20 dpm/mL.
- 7.21. Curium 244 tracer, NIST traceable, approximately 10-20 dpm/mL.
- 7.22. Neptunium 237 spike standard, 10-25 dpm/mL.

- 7.23. Plutonium 242 tracer (Pu-236 can also be used), NIST traceable, approximately 10-20 dpm/mL.
- 7.24. Plutonium 238 spike, NIST traceable, approximately 10-20 dpm/mL.
- 7.25. Plutonium 239/240 spike, NIST traceable, approximately 10-20 dpm/mL.
- 7.26. Thorium 229, NIST traceable 10-20 dpm/mL.
- 7.27. Natural Thorium spike standard (Th-232/Th-228), NIST traceable, approximately 10-20 dpm/mL.
- 7.28. Thorium 230 spike standard, NIST traceable, approximately 10-20 dpm/mL.
- 7.29. TRM solid reference material.
- 7.30. Uranium-232 tracer, NIST traceable, approximately 10-20 dpm/mL.
  - 7.30.1 Clean uranium is free of Th-228 daughter, removed by lead sulfate precipitation, activity verified prior to use.
  - 7.30.2 See SOP: ST-RC-0242 for the clean uranium procedure.

## 8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Samples may be collected in glass or plastic containers.
- 8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2, unless Iodine 129 or Iodine 131 is requested. Samples collected for I-129 or I-131 analysis are not preserved.
  - 8.3.1 The pH of aqueous samples are checked upon receipt by Sample Control, therefore, the pH does not require checking prior to analysis.
    - 8.3.1.1 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require checking the pH prior to analysis.

## 9.0 QUALITY CONTROL

- 9.1 **Batch**
  - 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same reagent lots. Where no preparation method exists (e.g. water samples for volatile organics analysis and inorganic anions analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, reagent lots and personnel.
  - 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
  - 9.1.3 For this analysis, batch QC consists of a method blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (DU). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
    - 9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.
  - 9.1.4 Samples having different QC criteria, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC jobs provided the actual "sample batch" does not exceed 20

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environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate job code.

**9.2 Method Blank (MB)**

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 For Oil analyses, the method blank is comprised of 1.25M Calcium Nitrate.
- 9.2.4 For Soil and other solid (non-filter) matrices, the method blank is comprised of 1.25M Calcium Nitrate.
- 9.2.5 For Filter analyses, the method blank is comprised of a blank filter, supplied either by the laboratory or the client.
- 9.2.6 For leached analyses, the method blank is comprised of the leaching acid.

**9.3 Laboratory Control Sample (LCS)**

- 9.3.1 A LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 A LCS must be prepared with every sample batch.
- 9.3.3 For Oil analyses, the LCS is comprised of 1.25M Calcium Nitrate fortified with the isotopes of interest.
- 9.3.4 For Soil, and other solid (non-filter) matrices, the LCS may consist of a solid reference material (e.g. TRM, NRM, PEM, or other) or 1.25M Calcium Nitrate fortified with the isotopes of interest.
  - 9.3.4.1 If Americium, Uranium, Curium and/or Plutonium are requested, fortify calcium nitrate with the requested isotopes.
  - 9.3.4.2 If Thorium is requested, either singly or in combination with other actinides, use the TRM (or other) reference material.
- 9.3.5 For Filter analysis, the LCS is comprised of a blank filter, supplied either by the laboratory or the client, fortified with the isotopes of interest.
- 9.3.6 For leached analyses, the LCS is comprised of the leaching acid, fortified with the isotope of interest.

**9.4 Matrix Spike (MS)/Matrix Spike Duplicate (MSD)**

- 9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.
- 9.4.3 MS/MSD samples, when requested, are performed with every sample batch and every LIMS batch.

**9.5 Sample Duplicate (DU)**

- 9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
- 9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of a LCSD for demonstration of precision.

**9.6 Procedural Variations/ Nonconformance and Corrective Action**

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

**10.0 CALIBRATION AND STANDARDIZATION**

- 10.1 Refer to the applicable analytical procedure for standardization of carrier/tracer solutions.
- 10.2 Balances and pipettes calibration must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipette".

## 11.0 PROCEDURE

- 11.1 This SOP addresses multiple sample preparation options based on the matrix and behavior of the samples. Samples are typically muffled prior to acid digestions unless the sample matrix is not conducive to muffling (e.g. high organic matter, construction debris). Samples are not typically muffled prior to sodium hydroxide fusions unless the sample matrix contains moderate to higher levels of organics and are conducive to muffling. Consult manager/supervisor prior to initiating sample preparation to determine which preparation is to be performed.
- 11.2 Soil and Sediment - Muffling
  - 11.2.1 Initiate sample preparation worksheet.
  - 11.2.2 Samples should be dried, ground, ball milled and/or pulverized as necessary, per ST-RC-0003.
  - 11.2.3 Pre-weigh a TEXPEN® labeled 50 mL glass beaker or zirconium crucible.
  - 11.2.4 Remove up to a 10 g aliquot for analysis, and place in the beaker or crucible. Record the exact weight of the sample taken to the nearest 0.0001 g in TALS. Weigh on an analytical balance.
    - 11.2.4.1 **The amount of sample required is determined by the reporting limit requested and can be up to 10 g. Usually a short count analysis requires a 1 g aliquot and a long count analysis requires a 2 g aliquot. These amounts are subject to change based on the client requirements, suspected activities of the isotopes, and count time requirements.**
  - 11.2.5 Prepare a Method Blank and LCS using 1.25M Calcium nitrate. Add enough to cover bottom of the muffling vessel.
  - 11.2.6 Wet samples with enough DI water to saturate the matrix.
  - 11.2.7 Spike the LCS and MS or MSD, with 10 - 40 dpm of the appropriate standard, for the requested target isotope(s).
    - 11.2.7.1 A soil reference material can be used for the LCS when the requested target isotopes are present.
    - 11.2.7.2 See Client Requirement sheet to determine which LCS is needed.
  - 11.2.8 Add 5 - 40 dpm of each tracer to all samples, blanks, LCS, and MS/MSD in the analytical batch that correspond to target isotopes requested.
    - 11.2.8.1 If samples are to be analyzed at a dilution, add the tracer after the digestions, unless client requirements specifically stipulate otherwise.
  - 11.2.9 Gently dry the samples on a hotplate so that it will not splatter.
  - 11.2.10 Place sample in the muffle oven and heat at 600°C for minimum of 4 hours.
    - 11.2.10.1 The TEXPEN® ink should turn white and become slightly ashy.
  - 11.2.11 Allow sample to cool to room temp.
- 11.3 Go to section 11.9 for acid digestion instructions or section 11.10 for alkaline fusion instructions.

#### Filters and Swipes - Muffling

**NOTE: The following procedure is for all filters that can be muffled safely via a ramped program without melting and binding to the crucible or beaker. This process is useful for filters (or swipes) that contain organic material as the high temperature of the muffle oven destroys such matter and allows for a much cleaner and simpler digestion. Plastic and Teflon filters should not be prepared using this method. Instead a leach should be performed. For these filter types proceed to section 11.8.**

**NOTE: If filter or swipes are to be split between analyses, spiking and tracing does not occur until after digestion.**

- 11.3.1 Label beakers with a TEXPEN® or metal marker to ensure identification post muffling.
- 11.3.2 Combine all paper filters which constitute the sample in a 250 mL glass beaker or a zirconium crucible.
- 11.3.3 Prepare a Method blank and LCS using the same number of clean, comparable filters.
  - 11.3.3.1 If no comparable filters are available, DI water blank can be used.
- 11.3.4 Spike LCS and, MS or MSD, with 10 - 40 dpm of appropriate standard, for the requested target isotopes.
  - 11.3.4.1 A soil reference material can be used for the LCS when the requested target isotopes are present.
  - 11.3.4.2 See Client Requirement sheet to determine which LCS is needed.
- 11.3.5 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch that corresponds to target isotopes requested.
- 11.3.6 Place sample beakers in muffle furnace and cover with a ribbed watch glass or zirconium cover as appropriate. If using zirconium cover, ensure the cover is slightly ajar to allow air flow into/out of the crucible.
  - 11.3.6.1 Ribbed watch glasses help ensure organic material is being destroyed.
- 11.3.7 Heat samples in a muffle furnace using the following sequence.
  - 11.3.7.1 Ramp the heat in the muffle furnace at 3° C a minute up to approximately 160° C.
  - 11.3.7.2 Heat samples for 45 minutes at approximately 160° C.
  - 11.3.7.3 Ramp the heat in the muffle furnace at 0.9° C per minute up to 600° C.
  - 11.3.7.4 Heat to approximately 600° C for 5-6 hours to reduce filters to ash.
- 11.3.8 If filters only partially reduced to ash, repeat steps 11.3.8.3 and 11.3.8.4, otherwise proceed
  - 11.3.8.1 NOTE: Not all filters will reduce to ash. As long as the organic material has lightened in color, samples can proceed to digestion or fusion.
  - 11.3.8.2 If filter has not reduced to ash after repetition and does not appear lightened in color, consult manager/supervisor for instruction.
- 11.3.9 Allow samples to cool to room temperature.
- 11.3.10 Proceed to section 11.9 for acid digestion instructions or section 11.10 for alkaline fusion instructions.

#### 11.4 Glass Fiber Filter Muffling Preparation

**NOTE: Glass fiber filters are ramped like others filters. Glass fiber filters also require a Hydrofluoric pre-digestion, which cannot take place in a glass beaker. Note – glass fiber filters may be fused directly (section 11.10) instead of acid digested with muffling if minimal organics are present. If muffling is required before fusing, do not perform 11.4.4-11.4.6**

**NOTE: If filter or swipes are to be split between analyses, spiking and tracing does not occur until after digestion.**

- 11.4.1 Refer to section 11.3.1 to 11.3.9 of this SOP for muffling instructions.
- 11.4.2 Transfer samples from glass beaker to Teflon beakers or digestion vessels. Rinse beaker with low molarity acid.

- 11.4.3 Place Teflon beakers on hotplate (temperature ~ 300° C). Digestion vessels will be placed in MOD block. (temp approximately 119°C)
- 11.4.4 Slowly add enough HF to cover sample. Acid should be added until the filter is dissolved.
  - 11.4.4.1 Caution: this is an extremely exothermic reaction.
- 11.4.5 Cook to dryness.
- 11.4.6 Proceed to section 11.9 for acid digestion instructions or section 11.10 for alkaline fusion instructions.
- 11.5 Flora and Fauna - Muffling
  - 11.5.1 Label beaker with is a TEXPEN® or metal marker to ensure identification post muffling.
  - 11.5.2 Weigh sample into beaker or a zirconium crucible and record.
  - 11.5.3 Prepare a Method Blank and LCS using 1.25M Calcium nitrate. Add enough to cover bottom of beaker.
  - 11.5.4 Spike LCS and, MS or MSD, with 10 - 40 dpm of standard, depending on the requested target isotopes.
    - 11.5.4.1 A soil reference material can be used for the LCS when the requested target isotopes are present.
  - 11.5.5 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch that corresponds to target isotopes requested.
    - 11.5.5.1 If samples are to be analyzed at a dilution, add the tracer after the dilution has been taken, unless client requirements specifically stipulate otherwise.
  - 11.5.6 Cover samples with concentrated nitric acid and allow samples to cook to dryness on a hotplate at low heat.
    - 11.5.6.1 This will help the samples break down.
    - 11.5.6.2 This converts samples to nitrates, which help destroy the sample in the muffle furnace in step 11.5.7.
  - 11.5.7 Place samples in muffle furnace and cover with a ribbed watch glass.
    - 11.5.7.1 Ribbed watch glasses help ensure organic material is being destroyed.
  - 11.5.8 Heat samples in a muffle furnace at approximately 600 ° C for minimum of 4 hours to reduce samples.
  - 11.5.9 Allow samples to cool to room temperature.
  - 11.5.10 Proceed to section 11.9 for acid digestion instructions or 11.10 for alkaline fusion instructions.
- 11.6 Oils, greases, and solvents - Muffling
  - 11.6.1 Label beakers with a TEXPEN® or metal marker to ensure identification post muffling.
  - 11.6.2 Add enough ashless media into the bottom of a beaker to absorb the oil and/or grease of the sample, and the tracer and spikes used.
  - 11.6.3 Weigh the sample into the beaker and cover with more ashless media.
  - 11.6.4 Prepare Blank and LCS crucible with the same amount of ashless media.
  - 11.6.5 For the LCS, MS, or MSD add 10-40 dpm of standard, depending on the requested isotopes.
  - 11.6.6 Add 5 - 40 dpm of each tracer to all samples and QC in the analytical batch that corresponds to target isotopes requested.
    - 11.6.6.1 If samples are to be performed at dilution, add the tracer after the dilution has been taken, unless client requirements specifically stipulate otherwise.
  - 11.6.7 Cover samples with a ribbed watch glass and heat in a muffle furnace using the following sequence.
    - 11.6.7.1 Ramp the heat in the muffle furnace at 3 ° C a minute up to approximately 160 ° C.
    - 11.6.7.2 Heat samples for 45 minutes at approximately 160 ° C.
    - 11.6.7.3 Ramp the heat in the muffle furnace at 0.9 ° C per minute up to 600 ° C.
    - 11.6.7.4 Heat to approximately 600 ° C for 5-6 hours to reduce samples.

- 11.6.8 Allow samples to cool to room temperature.
- 11.6.9 Proceed to section 11.9 for digestion instructions.
- 11.7 Wet Ashing – Non Muffling Preparation
  - 11.7.1 Wet Ashing is used for samples that were not muffled which have a noticeable amount of organics present.
  - 11.7.2 Initiate sample preparation worksheet.
  - 11.7.3 Aliquot an appropriate amount of sample into beaker.
    - 11.7.3.1 The amount of sample utilized is determined by the reporting limit and availability. Contact manager/supervisor for instruction.
  - 11.7.4 Weigh sample aliquot and record weight.
  - 11.7.5 Prepare a Method Blank and LCS using 1.25M Calcium nitrate. Add enough to cover bottom of beaker.
  - 11.7.6 Spike LCS and, MS or MSD, with 10 - 40 dpm of standard, depending on the requested target isotopes.
    - 11.7.6.1 A soil reference material can be used for the LCS when the requested target isotopes are present.
  - 11.7.7 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch that corresponds to target isotopes requested.
    - 11.7.7.1 If samples are to be performed at dilution, add the tracer after the dilution has been taken, unless client requirements specifically stipulate otherwise.
  - 11.7.8 Add 20 mL of concentrated Nitric acid enough to cover sample.
  - 11.7.9 Place samples on a hot plate on low heat so that sample does not splatter.
  - 11.7.10 Slowly add 1-2 mL of peroxide to the samples.  
**CAUTION: Samples can bubble vigorously.**
  - 11.7.11 Heat for 2- 5 minutes until bubbling slows.
  - 11.7.12 Repeat steps 11.7.11 and 11.7.12 until dark samples become lighter in appearance, indicating the destruction of the organics.
  - 11.7.13 Heat to dryness. Repeat 11.7.9 to 11.7.13 until dry sample becomes lighter in appearance.
  - 11.7.14 Proceed to section 11.9 for digestion instructions.
- 11.8 Leaching – Non Muffling Preparation
  - 11.8.1 The Leaching procedure is used for sample matrices not identifiably high in organic matter and not conducive to muffling. Method Blanks and QC samples are initiated after the leaching procedure.
  - 11.8.2 Aliquot an appropriate amount of sample into a labeled glass beaker and cover with a ribbed watch glass. Alternatively, the entire sample content may be acid leached. Contact manager/supervisor for instruction.
  - 11.8.3 Record sample weight on Sample Leach Log (Rad-0085).
  - 11.8.4 Add a known amount of nitric acid.
    - 11.8.4.1 The molarity of the acid will vary depending on the purpose of the leach and the matrix. If the purpose is a surface leach, the molarity of the nitric acid should be 1M.
  - 11.8.5 Record the amount of nitric acid used on the Sample Leach Log (RAD-0085).
  - 11.8.6 Allow sample to leach for a minimum of 4 hours.
  - 11.8.7 Cover Samples with a ribbed watch glass.
  - 11.8.8 Do not completely cover the sample, (allow gases to escape).
  - 11.8.9 Samples that do not totally dissolve need to be decanted into another labeled poly container.
  - 11.8.10 Aliquot the equivalent amount of leachate to represent 1-2 grams of sample.
    - 11.8.10.1 For example, if 50 grams of sample is leached with 500 mL of leaching solution, the aliquot would be 10-20 mL for each radiological analysis

requested. If all fields are filled in the Sample Leach Log, the targeted aliquot will be automatically calculated.

- 11.8.11 Prepare a Method Blank and LCS using 1.25M Calcium nitrate. Add enough to cover bottom of beaker.
  - 11.8.12 Spike LCS and, MS or MSD, with 10 - 40 dpm of standard, depending on the requested target isotopes.
    - 11.8.12.1 The MS/MSD is performed by taking an additional aliquot of leachate and fortifying it w/ the requested target isotopes.
  - 11.8.13 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch that corresponds to target isotopes requested.
  - 11.8.14 Proceed to section 11.9 for digestion instructions.
- 11.9 Mod Block or Teflon Beaker Total Digestion (Acid)
- NOTE: The amount of acid will vary depending on sample aliquot, when increasing the aliquot (5 g or more), increased acid amount will be required for total dissolution.**
- 11.9.1 Add approximately 5 mL of concentrated Hydrochloric acid to the beakers, cover with a watch glass, and allow to reflux for approximately 30 minutes.
    - 11.9.1.1 For samples that require dilutions, where there has been no spiking and tracing prior to muffling, the 5 mL reflux with HCL is not needed. Transfer samples into labeled digestion vessel using concentrated Nitric acid. Add approximately 5 mL of concentrated Hydrochloric Acid and proceed to 11.9.3.
  - 11.9.2 Transfer the samples to properly labeled mod block tubes or Teflon beakers with concentrated Nitric acid. Try to keep the total amount of acid around 5 mL.

**NOTE: Label the mod block tubes with a permanent black sharpie. Any other color ink degrades in the acid fumes and tubes will be unidentifiable.**
  - 11.9.3 Add approximately 10 mL of concentrated Hydrofluoric acid.
  - 11.9.4 Place digestion vessels into MOD block and set temperature to 119°C. Allow samples to go to dryness. If teflon beakers are being used, heat on hot plate so that samples do not splatter. Allow samples to go to dryness (entire step is called 1<sup>st</sup> digestion).
  - 11.9.5 Add approximately 5 mL of concentrated HNO<sub>3</sub>, 5 mL of concentrated HCl and 10 mL of concentrated HF to digestion vessels/teflon beakers. Allow the samples to go to dryness (step is 2<sup>nd</sup> digestion).
  - 11.9.6 For samples that are being split with other analysis, proceed to 11.9.7. For all other samples, proceed to 11.9.9.
  - 11.9.7 Add enough 1M or 3M HNO<sub>3</sub> to cover sample and allow to reflux for approximately 30 minutes with a watch glass over the top of the vessel/beaker.
  - 11.9.8 Transfer to a 50 or 100 mL digestion vessel. Dilute sample and record volume on cap of tube.
    - 11.9.8.1 Aliquot will depend on reporting limit and analyses requested on sample. Please consult supervisor or PM.
    - 11.9.8.2 For samples that are being split with other analysis, place samples aliquot into a new, labeled digestion vessel. Allow samples to go to dryness first before moving on to 11.9.9.
  - 11.9.9 Add 5 mL of 3M HNO<sub>3</sub> and 0.25M Boric Acid to each sample
    - 11.9.9.1 For samples that required a dilution, place sample aliquot into a new labeled digestion vessel. Allow samples to go to dryness first before adding 5 mL of 3M HNO<sub>3</sub> and 0.25M Boric Acid.
  - 11.9.10 Add 10 mL of Load solution, cover with a watch glass, and reflux for 20-30 minutes (Step is Boric/Load ).
  - 11.9.11 Using 3M nitric acid, transfer samples to properly labeled centrifuge tubes.
    - 11.9.11.1 For samples requiring a dilution, transfer sample (dissolved in 10 mL of load solution) to a labeled 50 or 100 mL digestion vessel with 3M nitric acid.
    - 11.9.11.2 Dilute the sample to a known volume with 3M HNO<sub>3</sub>. Record this volume on the cap of the digestion vessel.

- 11.9.11.3 Aliquot the proper amount of sample needed to achieve the dilution into a labeled centrifuge tube.
- 11.9.11.4 If sample aliquot is less than 5 mL, then bring the volume up to 15 mL with load solution. If aliquot is greater than 5 mL, then the sample will have to be cooked to dryness prior to adding load solution.
- 11.9.12 Proceed directly to the appropriate column SOP.
- 11.10 Acid Digestion (Nitric/Hydrochloric – aqua regia)
  - 11.10.1 This digestion is intended for soil samples that are expected to contain nuclides in a reasonably soluble state. If the sample is expected to contain analytes of interest in a refractory or otherwise relatively intractable matrix (e.g. trapped in silicon crystalline structure), a total digestion or alkaline fusion may be more appropriate. Consult your supervisor or technical director if necessary.
  - 11.10.2 This digestion may be performed on dried/disaggregated or “as-received” samples.
    - 11.10.2.1 For dried/disaggregated samples, prepare sample as per SOP ST-RC-0003, “Drying and Grinding of Soil and Solid Samples”.
  - 11.10.3 Weigh samples into a labeled crucible (if muffling is necessary – see 11.10.5 below) or directly into a digestion tube or beaker (if muffling is not necessary). A typical aliquot will be 1 g. Smaller aliquots (down to 0.25 g) may be used due to matrix issues. Larger aliquots (up to 10 g) may be utilized but acid volumes may need to be adjusted accordingly. See your supervisor or technical director for instructions.
  - 11.10.4 Add appropriate carrier(s)/tracer(s) per the separation method SOP. Add spike(s) to the LCS and MS/MSD, if applicable.
  - 11.10.5 If the sample is known or suspected to contain a significant amount of organic matter (or if you are unsure about the organic content):
    - 11.10.5.1 If the sample was previously dried/disaggregated:
      - 11.10.5.1.1 Place the sample briefly on a hotplate to take the carrier/tracer, spike, or any other reagents to dryness. Do not allow the sample to spatter.
      - 11.10.5.1.2 Place the sample in an oven at 600° and allow to muffle for 4 hours. Allow to cool.
    - 11.10.5.2 If the sample is to be analyzed on an “as-received” basis:
      - 11.10.5.2.1 Ramp the sample slowly in an oven, ultimately reaching 600° for 4 hours. Allow to cool.
  - 11.10.6 If the sample was muffled and is to be digested in a vessel other than the crucible:
    - 11.10.6.1 Add about 5 mL of concentrated hydrochloric acid to the crucible and reflux covered with a watch glass for about 30 minutes. Transfer the soil to a labeled **(use black permanent marker – other colors may degrade in the acid fumes)** digestion tube or beaker. Rinse the crucible with 5 mL concentrated nitric acid. Add 10 mL more nitric acid to the digestion vessel.
  - 11.10.7 If the sample was not muffled or will be digested directly in the crucible:
    - 11.10.7.1 Carefully add 15 mL concentrated nitric acid and 5 mL concentrated hydrochloric acid to the sample.
  - 11.10.8 Digest in mod block (digi-tube) or on hot plate (beaker) at > 110° for 1-2 hours. If on a hot plate, place a watch glass on the beaker. Mix/agitate the digestion vessel periodically.
  - 11.10.9 Allow the solution to cool.
  - 11.10.10 Transfer the solution/solids to a labeled centrifuge tube with minimal DI water and centrifuge. Carefully transfer the supernatant to a labeled beaker.
  - 11.10.11 Add 10 mL of concentrated nitric acid to the solids remaining in the centrifuge tube and vortex or mix to loosen the solids. Transfer the solids/solution to the original digestion vessel. Rinse the tube with 5 mL concentrated nitric acid to ensure all the soil transfers, and add to the digestion vessel. Add 5 mL concentrated hydrochloric acid to the digestion vessel.

- 11.10.12 Digest in mod block (digi-tube) or on hot plate (beaker) at  $> 110^{\circ}$  for 1-2 hours. If on a hot plate, place a watch glass on the beaker.
- 11.10.13 Transfer the solution /solids to a labeled centrifuge tube with minimal DI water and centrifuge. Carefully transfer/combine the supernatant with the supernatant from the first digestion. Discard the tube with the residual soil appropriately.
- 11.10.14 Carefully evaporate the combined supernatant to near dryness (don't allow the sample to go completely dry, especially if the analyte(s) of interest may become volatilized).
- 11.10.15 Proceed to the appropriate separation SOP.
- 11.11 Alkaline Fusion (Sodium Hydroxide)
  - 11.11.1 Soil and/or glass fiber filter matrix samples may be fused without prior muffling if the samples do not contain significant organic matter. Otherwise, matrices should be muffled first (see above appropriate steps) to destroy the majority of organics in the sample.
  - 11.11.2 If the sample was previously muffled in a zirconium crucible, skip to step 11.10.11.
  - 11.11.3 Initiate sample preparation worksheet.
  - 11.11.4 Soil samples should be dried, ground, ball milled and/or pulverized as necessary, per ST-RC-0003.
  - 11.11.5 Pre-weigh a labelled zirconium crucible.
  - 11.11.6 Remove an aliquot for analysis (see 11.2.4.1 for guidance, or contact your supervisor). Weigh on an analytical balance and record the exact weight to the nearest 0.0001 g in TALS.
  - 11.11.7 Prepare a Method Blank and LCS using 1.25M calcium nitrate. Add enough to cover the bottom of the beaker or crucible.
    - 11.11.7.1 A soil reference material may be used for the LCS when the requested target analytes are present at appropriate concentration. If such a reference material is used, do not add calcium nitrate at this step.
  - 11.11.8 Spike the LCS (and MS/ MSD if required) with 10-40 dpm of the appropriate standard(s) for the requested target isotope(s).
    - 11.11.8.1 See client requirement sheet or backlog to determine which isotopes are desired. If a soil reference material is used for the LCS, do not spike analytes contained in the reference material.
  - 11.11.9 Add 5-40 dpm of each tracer to all samples, MB, LCS, and MS/MSD in the analytical batch that correspond to the target isotopes requested.
    - 11.11.9.1 If samples are to be analyzed at a dilution, add the tracer after the fusion, unless client requirements specifically stipulate otherwise.
  - 11.11.10 Gently dry the samples on a hotplate so they don't splatter. Failure to bring to dryness could cause creeping or popping of samples during the fusion process.
  - 11.11.11 Allow to cool slightly, and then add 10 g of sodium hydroxide (solid) to each crucible for each 1 g of sample.
  - 11.11.12 Cover the crucibles with zirconium lids, and muffle at  $600^{\circ}\text{C}$  for approximately 30 minutes.
    - 11.11.12.1 While the crucibles can be placed into a pre-heated muffle oven, starting with a room temperature oven may help prevent creeping and/or popping of the sample.
    - 11.11.12.2 The TEXPEN® ink should turn white and become slightly ashy.
  - 11.11.13 Allow the crucibles to cool enough to be able to handle safely.
  - 11.11.14 Add 125 mg (2.5 mL of 50 mg/mL) iron (as ferric nitrate) and 5 mg lanthanum (0.5 mL of 10 mg/mL) lanthanum (as lanthanum nitrate) to 225 mL centrifuge tubes. This can be done in advance to save time.
  - 11.11.15 Move the crucibles to a hot plate, and using DI water with heating transfer the solids to the 225 centrifuge tubes. Add additional DI water to the crucibles and heat as needed to remove the residual solids.



- 11.11.16 Rinse crucibles with 10 mL hot 3M HNO<sub>3</sub> (helps remove uranium and thorium from the crucible).
- 11.11.17 Dilute to approximately 175 mL with DI water, cool to room temperature in ice bath.
- 11.11.18 Add 2 mL of 1.25M Ca(NO<sub>3</sub>)<sub>2</sub> and 3 mL of 3.2M (HN<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> and mix well (calcium and phosphate ions enhance uranium recovery).
- 11.11.19 Add 5 mL of 20% titanium trichloride and mix well (reduces U(VI) to U(IV) to improve U recovery during precipitation steps). Cool in ice bath to room temperature (~10 min).
- 11.11.20 Centrifuge at 3500 rpm for 5 min (or 2000 rpm for 8-10 min), and then discard the supernatant.
- 11.11.21 Dissolve the remaining precipitate as much as possible in a total volume of ~80 mL of 1.5 M HCl.
- 11.11.22 Dilute to ~175 mL with 0.01 M HCl.
- 11.11.23 Add 1 mg of lanthanum (0.1 mL of 10 mg/mL lanthanum nitrate) and 25 mg Ca (0.5mL of 1.25M Ca(NO<sub>3</sub>)<sub>2</sub>) to each sample.
- 11.11.24 Add 3 mL of 20% titanium trichloride to each sample; mix (to ensure no actinides are in the hexavalent state and to facilitate complete precipitation).
- 11.11.25 Add 10-25 mL of 28M hydrofluoric acid to each sample; mix well. This will dissolve more of the iron hydroxide/titanium hydroxide precipitate and cause a smaller lanthanum/calcium fluoride precipitate to form.
- 11.11.26 Place on ice for ~10 minutes (to reduce solubility). Then allow to stand ~5 minutes.
- 11.11.27 Centrifuge at 3500 rpm for 10 minutes (or 2000 rpm for 10 min), and then discard the supernatant. (Note that the LaF<sub>3</sub> removal step effectively removes almost all of the Fe and Ti, as well as silicates that can affect column flow.)
- 11.11.28 Dissolve/transfer the residual solids containing the actinides with 10 mL of warm 3M HNO<sub>3</sub> – 0.25 M boric acid into a 50mL centrifuge tube. Rinse the 225mL centrifuge tube with 6 mL of 7M HNO<sub>3</sub>, and then 8.5 mL of 2 M aluminum nitrate (scrubbed to remove trace U), transferring each to the 50mL tube. Mix (e.g. vortex) the samples and heat for 2-5 minutes in a 80 °C water bath to help dissolve the solids.
- 11.11.29 The tubes may be centrifuged to check for any traces of solid particulates. If found, the particulates may be removed (either by decanting the solution or by filtering the sample to a new tube).
- 11.11.30 Add 1mg of Fe (20 uL of 50 mg/mL or 200uL of 5 mg/mL iron nitrate) and 1.3 mL of 1.4 M ascorbic acid; mix. Wait ~3 minutes or more before the next step. The ascorbic acid should be made fresh. This allows plutonium to be reduced to Pu(III).
- 11.11.31 Add 1 mL of 3.5 M sodium nitrite (0.24g to 25 mL total w/DI); mix. (to oxidize Pu to +4)
- 11.11.32 Add 1.5 mL 15.8 M nitric acid (enhances Am/Cm retention).
- 11.11.33 Proceed directly to the appropriate column SOP.

## 12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 There are no calculations pertaining to this sample preparation procedure.
- 12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis Quality Manual (ST-QAM). Specific analysis calculations are given in the applicable analysis SOP.

### **13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA**

- 13.1 Data assessment does not pertain to this sample preparation procedure.
- 13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

### **14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY**

- 13.1. Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS.
- 13.2. Demonstration of Capability
  - 13.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 13.3. Training Qualification
  - 13.3.1. The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
  - 13.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 13.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

### **15.0 VALIDATION**

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

### **16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION**

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
  - 16.2.1 The following waste streams are produced when this method is carried out.
    - 16.2.1.1 Acidic sample is waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
    - 16.2.1.2 Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
    - 16.2.1.3 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

## 17.0 REFERENCES

- 17.1 TestAmerica Quality Assurance Manual (ST-QAM), current revision.
- 17.2 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
- 17.3 Associated SOPs:
  - 17.3.1 ST-PM-0002, "Sample Receipt and Chain-of-Custody."
  - 17.3.2 ST-QA-0002, "Standards and Reagent Preparation."
  - 17.3.3 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and pipettes."
  - 17.3.4 ST-QA-0036, "Non-conformance Memorandum (NCM) Process."
  - 17.3.5 ST-RC-0003, "Drying and Grinding of Soil and Solid Samples."
  - 17.3.6 ST-RC-0100, "Actinide Coprecipitation."
  - 17.3.7 ST-RC-0232, "Isotopic Thorium and/or Neptunium in Water, Soil, Sludge, and Filters by Eichrom® Teva Separation Resin."
  - 17.3.8 ST-RC-0238, "Isotopic Uranium by Eichrom® Uteva Resin For Water, Soil, Sludge and Filters."
  - 17.3.9 ST-RC-0240, "Isotopic Americium, Curium, Plutonium, Thorium, and Uranium in Water, Soil, Sludge and Filters by Eichrom® Separation Resin."
  - 17.3.10 ST-RC-0241, "Americium, Plutonium, Curium and Uranium in Water, Soil, Sludge and Filters by Eichrom® Uteva and Tru resins."
  - 17.3.11 ST-RC-0242, "Isotopic Thorium and Uranium in Water, Soil, Sludge and Filters by Eichrom® Separation Resins."

## 18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 None.

## 19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 The removal of section 11.6.8 regarding hotplates in section 11.0
- 19.2 Rev. 18:
  - 19.2.1 In section 9.2, removed analysis reference for "other solids and non-filters" and inserted instructions for leached analysis.
  - 19.2.2 Changed the normality of nitric acid needed for a surface leach from 2M to 1M in section 11.8.4.1.1 and removed the stipulation regarding using the 1M solution if there are not heavy metals present.
- 19.3 Rev. 19:
  - 19.3.1 In section 11.9.5, added a note stating the amount of acid is increased as aliquot size is increased for total dissolution.
- 19.4 Rev. 20:
  - 19.4.1 Updated equipment in section 6.
  - 19.4.2 Updated Nitric Acid and Uranium-232 tracer in section 7.
  - 19.4.3 Updated preservation requirements and added pH checks to section 8.
  - 19.4.4 Updated muffling procedure in section 11, as well as wet ashing and leaching.
  - 19.4.5 Removed uranium from section 9.3.4.2.
  - 19.4.6 Updated instructions for preparing Method Blanks throughout section 11.0.
  - 19.4.7 Updated instructions for MOD Block Digestion in section 11.9.
- 19.5 Rev. 21:

- 19.5.1 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.
- 19.5.2 Removed references to ‘Clouseau’ and ‘QuantIMS’ and replaced them with ‘LIMS’.
- 19.5.3 Updated section 7.0. by adding new dilution ratio’s for Boric Acid.  
Updated Mod Block Total Digestion in section 11.9.  
Updated section 15.0
- 19.6 Rev. 22: (07/31/2015)
  - 19.5.4 Section 5.0 updated – perchloric acid removed
  - 19.5.5 Section .6.0 updated – equipment related to use of perchlorate acid removed
  - 19.5.6 Section 7.0 updated – concentration of HNO<sub>3</sub> updated
  - 19.5.7 Section 9.0 updated – uranium added to Section 9.3.4.1
  - 19.5.8 Procedure updated in Section 11.0 – perchloric acid prep references removed
  - 19.5.9 Grammatical errors fixed through out SOP
- 19.6 Rev. 23: (06/22/2015)
  - 19.6.6 Grammatical errors fixed through out SOP
  - 19.6.7 Updated Section 11.0
  - 19.6.8 Added Sample Leach Log (Rad-0085) and names of digestion steps  
Annual Review, No Changes (05/23/2016)
- 19.7 Revision 24: (04/21/2017)
  - 19.7.6 Updated section 5.0: corrected acronym from MSDS to SDS
  - 19.7.7 Updated section 7.0 : clarified how to make reagents
  - 19.7.8 Updated section 9: change acronym for sample duplicated, from SD to DU, to match LIMS
- 19.8 Revision 25 (06/30/2017)
  - 19.8.1 Updated material to use for solid LCS
  - 19.8.2 Added sodium hydroxide fusion prep to SOP
  - 19.8.3 Clarifications added to sections dealing with muffling
- 19.9 Revision 26 (03/07/2018)
  - 19.9.1 Technical Review S. Bernsen; QA Review M. Ward
  - 19.9.2 Updated section 1.2- added “a portion of sample”
  - 19.9.3 Updated section 11.8.2- removed “500 mL of 1 L”. Added “ribbed watchglass”.
  - 19.9.4 Updated section 11.9.9.1- removed, “cover and reflux for 10-15 minutes until solids have fully dissolved”.
- 19.10 Revision 27 07/17//2018) Technical Review T. Romanko/QA Review M Ward
  - 19.10.1 Inserted section 11.10 - Acid Digestion (Nitric/Hydrochloric – aqua regia)
  - 19.10.2 Updated title to be applicable to any Radiochemical analysis.
  - 19.10.3 Updated sections 1 and 2 to apply to any Radiochemical analysis.
- 19.11 Revision 28 (09/23/2019) Technical Review –C. Mazariegos, QA Review – K. Ely
  - 19.11.1 Added Eurofins logo and updated copyright information.
  - 19.11.2 Removed references to ST-RC-0014 archived SOP.
  - 19.11.3 Section 9 – removed duplicate paragraph
  - 19.11.4 11.6.7 clarified requires the use of a ribbed watch glass.



Environment Testing  
TestAmerica





TestAmerica St. Louis

SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 1 of 13

## Title: RADIUM-226 AND RADIUM-228 BY CHEMICAL SEPARATION PREPARATION

Approvals (Signature/Date):	
 Chelsea Mazariegos Radiochemistry Prep Supervisor	 Michael Ridenhower Health & Safety Manager / Coordinator
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This SOP was previously identified as SOP No. ST-RC-0041 Rev. 17

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### 1.0 SCOPE AND APPLICATION

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 2 of 13

- 1.1 This method covers the chemical separation preparation for radium-226 and radium-228 by direct measurement of its beta emitting progeny, Actinium ( $^{228}\text{Ac}$ ). It is applicable to liquid (e.g water and wastewater) where complete dissolution and carrier exchange are readily achievable in the laboratory. For media where chemical dissolution is impractical, non-destructive measurement of the three principal photons of  $^{228}\text{Ac}$  by gamma spectrometry is better suited.
- 1.2 This SOP is based on EPA Method 904.0, 903.0, SW9315, and SW9320.
- 1.3 The barium sulfate precipitate from this procedure contains all radium isotopes.
- 1.4 The barium sulfate can be counted for total alpha radiation. The time of the last barium sulfate precipitation should be recorded and used in calculating the in-growth factor.
- 1.5 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

## 2.0 SUMMARY OF METHOD

- 2.1 Radium isotopes are collected by coprecipitation with barium and lead sulfate and purified by precipitation from EDTA solution. After a suitable ingrowth period,  $^{228}\text{Ac}$  is separated and carried on yttrium oxalate, purified and counted for the presence of total beta radiation. The precipitation and counting are performed in a manner consistent with the time requirements of the 6.13 hour half life of  $^{228}\text{Ac}$ . By applying correction factors for ingrowth and decay and appropriately calibrating the beta counter, radium-228 is quantified. The barium sulfate fraction, minus radium-224, is counted by Gas Flow Proportional Counting (GFPC) to report radium-226.

## 3.0 DEFINITIONS

- 3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data reporting qualifiers.
- 3.2 There are no specific definitions for this procedure.

## 4.0 INTERFERENCES

- 4.1 Strontium 90 or other beta emitting radionuclides that are carried by the yttrium oxalate precipitate (i.e. certain mixed fission or activation products) will yield a positive bias to the radium-228 values.
- 4.2 Samples which contain natural barium cause inaccurate chemical yield determinations.

## 5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
  - 5.2.1 None.

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SOP No. ST-RC-0041, Rev. 18  
Effective Date: 03/08/2019  
Page No.: 3 of 13

### 5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the SDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

Material <sup>(1)</sup>	Hazards	Exposure Limit <sup>(2)</sup>	Signs and symptoms of exposure
Ammonium Hydroxide	Poison Corrosive	50 ppm	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Acetic Acid, Glacial	Corrosive Flammable	10 ppm (TWA)	Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of vision.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm- (TWA) 4 ppm- (STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M <sup>3</sup> - (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

## 6.0 EQUIPMENT AND SUPPLIES

- 6.1 Centrifuge tubes, 50-mL
- 6.2 Centrifuge
- 6.3 Hot Plate
- 6.4 Analytical balance (four decimal places)

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 4 of 13

- 6.5 Stainless steel planchets
- 6.6 Beakers, various volumes
- 6.7 Syringe, 20-mL, 50 mL, 60 mL
- 6.8 Water Bath
- 6.9 Desiccator
- 6.10 Pipette

## 7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.
- 7.2 DI water
- 7.3 Acetic acid, 17.4 M: glacial  $\text{CH}_3\text{COOH}$  (concentrated), specific gravity 1.05, 99.8%.
- 7.4 Ammonium hydroxide, 15 M:  $\text{NH}_4\text{OH}$  (concentrated), sp. gr. 0.90, 56.6%.
- 7.5 Ammonium oxalate, 5%: non-critical reagent Dissolve approximately 2 g  $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$  in water and dilute to approximately 40 mL.
- 7.6 Ammonium sulfate, 200 mg/mL: non-critical reagent: dissolve approximately 400 g  $(\text{NH}_4)_2\text{SO}_4$  in water and dilute to approximately 2000 mL.
- 7.7 Ammonium sulfide, 2%: Dilute 5 mL  $(\text{NH}_4)_2\text{S}$ , (20-24%), to 50 mL water; total volume 50 mL
- 7.8 CPI Barium carrier (standardized), 33.9 mg/mL,  $\text{BaSO}_4$  (20 mg/ml Ba)
  - 7.8.1 If the barium carrier is not already standardized(from CPI) , standardize the barium carrier solution using the following procedure.
    - 7.8.2 Pipette 1.0 mL barium carrier solution (20 mg/mL, Ba) into six separate labeled centrifuge tubes containing 15 mL DI  $\text{H}_2\text{O}$ .
    - 7.8.3 To each tube, add 1 mL 18N sulfuric acid while stirring and digest precipitate in a hot water bath for approximately 10 min.
    - 7.8.4 Cool, centrifuge and decant the supernate into appropriate waste container.
    - 7.8.5 Wash precipitate with 15 mL DI water, centrifuge and decant the supernate.
    - 7.8.6 Transfer the precipitate to a pre-weighed stainless steel planchet with a minimal amount of DI water.
    - 7.8.7 Dry on a heat source. Store in desiccator until cool and weigh as barium sulfate.
      - 7.8.7.1 Record the net weights of the precipitates and calculations in the Rad Standards Preparation Log.
- 7.9 Citric acid, 1M: non-critical reagent: Dissolve approximately 192 g  $\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$  in water and dilute to approximately 1000 mL.
- 7.10 EDTA reagent basic (0.25M)non-critical reagent - dissolve approximately 20 g NaOH in approximately 750 mL water, heat and slowly add approximately 93 g [ethylenedinitrilo] tetraacetic disodium salt,  $(\text{C}_{10}\text{H}_{14}\text{O}_8\text{N}_2\text{Na}_2 \cdot 2\text{H}_2\text{O})$  while stirring. Dilute to approximately 1 L.

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 5 of 13

- 7.11 Lead carrier, 15 mg/mL:non-critical reagent: Dissolve approximately 23.97 g  $\text{Pb}(\text{NO}_3)_2$  in water, add approximately 5 mL of 6M  $\text{HNO}_3$  and dilute to approximately 1000 mL with water.
- 7.12 Lead carrier, 1.5 mg/mL:non-critical reagent: Dilute approximately 10 mL lead carrier, (15 mg/mL), to approximately 100 mL with water.
- 7.13 Methyl orange indicator, 0.1%:non-critical reagent: Dissolve approximately 0.1g methyl orange indicator in approximately 100 mL water.
- 7.14 Nitric acid, 16M:  $\text{HNO}_3$  (concentrated), specific gravity 1.42, 70.4%.
- 7.14.1 Nitric acid, 6M: non-critical reagent: Mix approximately 3 volumes 16M  $\text{HNO}_3$  (concentrated) with approximately 5 volumes of water.
- 7.14.1.1 Nitric acid, 2N: non-critical reagent: Mix approximately 1 volume 6N  $\text{HNO}_3$  with approximately 2 volumes of water.
- 7.15 Sodium hydroxide, 18N:non-critical reagent:Dissolve approximately 72g NaOH in water and dilute to approximately 100 mL.
- 7.16 Sodium hydroxide, 10N: non-critical reagent: dissolve approximately 40g NaOH in water and dilute to approximately 100 mL.
- 7.17 Strontium carrier, 10 mg/mL: non-critical reagent: Dissolve approximately 24.16 g  $\text{Sr}(\text{NO}_3)_2$  in water and dilute to approximately 1 liter.
- 7.18 Sulfuric acid, 18 N:non-critical reagent: cautiously mix approximately 1L 36N  $\text{H}_2\text{SO}_4$  (concentrated) with approximately 1L of water
- 7.19 Yttrium carrier(standardized), 5 mg/ml
- 7.19.1 Yttrium carrier from CBI- 50 mg/mL
- 7.19.1.1 If the yttrium carrier is not already standardized, standardize the yttrium carrier solution using the following procedure.
- 7.19.1.2 Prepare 10 samples of the carrier solution. Perform the following steps on each vial.
- 7.19.1.3 Add 5 mL DI water to a 50 mL centrifuge tube.
- 7.19.1.4 Add 2 mL concentrated nitric acid.
- 7.19.1.5 Add 1 drop of MCP indicator (0.1%).
- 7.19.1.6 Add 1.0 mL of yttrium carrier (10 mg/mL, Y) pipette.
- 7.19.1.7 Add 5 mL of 0.4 M oxalic acid.
- CAREFULLY add concentrated ammonium hydroxide dropwise until a reddish yellow end point is obtained.**
- NOTE: The pH must be between 1.7 - 1.9 to assure a uniform  $9\cdot\text{H}_2\text{O}$  hydrate yttrium oxalate precipitate. The yttrium oxalate will have precipitated.**
- 7.19.1.8 Heat the solution for approximately 5 –10 minutes in a hot water bath.
- 7.19.1.9 Cool the solution for approximately 5 – 10 minutes in an ice water bath.
- 7.19.1.10 Centrifuge.
- 7.19.1.11 Decant supernate into the appropriate waste container.
- 7.19.1.12 Add approximately 20 mL of DI water to the precipitate.
- 7.19.1.13 Vortex and centrifuge.
- 7.19.1.14 Decant supernate into the appropriate waste container.
- 7.19.1.15 Add approximately 20 mL of DI water to the precipitate.
- 7.19.1.16 Vortex and Centrifuge.
- 7.19.1.17 Decant supernate into the appropriate waste container.

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 6 of 13

- 7.19.1.18 Add approximately 5 mL of D.I. water, slurry the precipitate.
- 7.19.1.19 Pre-weigh a planchet and record the tare weight on the sample worksheet.
- 7.19.1.20 Transfer the slurry to the planchet.
- 7.19.1.21 Dry the planchet on a hotplate.
- 7.19.1.22 Remove from heat and cool to room temperature in a desiccator.
- 7.19.1.23 Weigh sample to determine yttrium yield.
- 7.19.1.24 Repeat heating and cooling in the desiccator until a constant weight is obtained as determined by two consecutive measurements where the weight differences are  $\pm 5\%$  or less.
- 7.19.1.25 Record gross and final weights in LIMS. .
- 7.20 Yttrium carrier, -9 mg/mL: Dilute 50 mL yttrium carrier, to 100 mL with water.
- 7.21 Strontium-yttrium mixed carrier, 0.9 mg/mL  $\text{Sr}^{+2}$ ; 0.9 mg/mL  $\text{Y}^{+3}$ :
  - 7.21.1 Dissolve 2.175 g of  $\text{Sr}(\text{NO}_3)_2$  in 200 mL of DI water. Add 90 mL of 50 mg/mL Y carrier. Dilute to final volume of 500 mL
- 7.22 Radium-226, standard 20-25 dpm
- 7.23 Radium-228, standard 20-25 dpm

## 8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in ST-PM-0002.
- 8.2 Samples may be collected in glass or plastic containers.
- 8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

## 9.0 QUALITY CONTROL

### 9.1 Batch

- 9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch comprises of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
  - 9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.
- 9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

### 9.2 Method Blank

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 7 of 13

- 9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
- 9.2.2 A method blank must be prepared with every sample batch.
- 9.2.3 For Liquid analyses, the method blank is comprised of DI water acidified with 2 mL of nitric acid.
- 9.2.4 For Soil analyses, the method blank is comprised of DI water acidified with 2 mL of nitric acid.

**9.3 Laboratory Control Sample**

- 9.3.1 An LCS is a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every sample batch.
- 9.3.3 For Liquid analyses, the LCS is comprised of DI water fortified with radium-226 and radium-228.
- 9.3.4 For Soil analyses, the LCS is comprised of radium-226 and radium-228.

**9.4 Matrix Spike/Matrix Spike Duplicate**

- 9.4.1 A Matrix Spike (MS) is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch. MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

**9.5 Sample Duplicate**

- 9.5.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.  
If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed. A NCM is written to document the insufficient volume and utilizing of an LCSD for demonstration of precision.

**9.6 Procedural Variations/ Nonconformance and Corrective Action**

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.
- 9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

**10.0 CALIBRATION AND STANDARDIZATION**

- 10.1 Balance and thermometer calibration must be checked daily when used. Refer to SOP ST-QA-0005.
- 10.2 See the analytical SOP for instrument calibration; ST-RD-0403

**11.0 PROCEDURE**

- 11.1 Water Samples
  - 11.1.1 Ensure that the sample container is capped tightly and shake it thoroughly. Ensure that the sample is at or below a pH of 2. Transfer a sample aliquot to a beaker.
  - 11.1.2 Sample aliquot size is 1 liter.

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 8 of 13

- 11.1.2.1 For client requesting a reporting limit less than 1pCi/L, a larger sample volume may be required. Contact Radiochemistry manager/supervisor for instruction.
- 11.1.2.2 If less than 1 liter of sample was provided by the client, write the NCM noting insufficient volume.

## 11.2 Soil Samples

- 11.2.1 For soil samples prepare per SOP ST-RC-0003, "Drying and Grinding of Soil and Solid Samples", and weigh 1 to 2 grams into a labeled crucible.
- 11.2.2 Place in oven at 600 °C and allow to muffle for four hours. Allow to cool.
- 11.2.3 Transfer to digestion tube, or Teflon beaker using 4M HNO<sub>3</sub>.
- 11.2.4 Add 1 mL of standardized barium carrier to samples and QC. Add radium-226 and radium-228 spike to LCS and MS/MSD, if applicable.
- 11.2.5 Carefully add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.
- 11.2.6 Digest in mod block at >110°, or on a hotplate for four hours or until dry.
- 11.2.7 Carefully add 10 mL concentrated nitric acid, 10 mL concentrated hydrochloric acid and 5 mL concentrated Hydrofluoric acid.
- 11.2.8 Digest in mod block at >110°, or on a hotplate for four hours or until dry.
- 11.2.9 Dissolve with 10 mL HNO<sub>3</sub> and 10 mL HCl, return to mod block, or hotplate for 30min.
- 11.2.10 Transfer to 400 mL beakers with 4M HNO<sub>3</sub>. Dilute to 200 mL with DI water.

## 11.3 Initial Precipitation

- 11.3.1 Add methyl orange indicator until pink endpoint persists. For soils skip to 11.3.4.
- 11.3.2 Add 1.0 mL standardized barium carrier (33.9 mg/mL).
- 11.3.3 Spike LCS and MS/MSD (if applicable) with radium-226 and radium-228.
- 11.3.4 Add 1M citric acid in ratio of 5 mL per liter. Mix thoroughly.
- 11.3.5 Add 2.5 mL lead carrier (15 mg/mL), 2 mL strontium carrier (10 mg/mL), and 0.2 mL yttrium carrier (9 mg/mL); stir well. (mass = 0.0249 g)
- 11.3.6 Slowly add 15M ammonium hydroxide until a definite yellow color is obtained, then add a few drops more. If no yellow endpoint is visible, insure pH is > 6.5.
- 11.3.7 Stir and heat until close to boiling for about 30 minutes.
- 11.3.8 **Face shield must be worn during this process.** Precipitate lead and barium sulfates by adding 10 mL of 18N sulfuric acid, or until the pink endpoint reappears.
- 11.3.9 Add 5 mL ammonium sulfate (200 mg/mL) for each liter of sample. Stir frequently and keep at a temperature of approximately 90°C for 30 minutes until a barium sulfate precipitate forms.
- 11.3.10 Allow precipitate to settle to the bottom of the beaker for a least 6 hours.

## 11.4 Reprecipitation Clean up / Ac-228 Ingrowth

- 11.4.1 Decant the supernate and discard to acid waste, taking care to avoid disturbing the precipitate.  
Transfer precipitate to a 50 mL centrifuge tube, taking care to rinse last particles out of beaker with DI water. Centrifuge and discard supernate.
- 11.4.2 Wash the precipitate with 10 mL 16M HNO<sub>3</sub>, vortex, centrifuge, and discard supernate to acid waste. Repeat this step.
- 11.4.3 Wash the precipitate with 10 mL D.I.H<sub>2</sub>O, vortex, centrifuge, and discard supernate to acid waste. Repeat this step.
- 11.4.4 Add 1 mL strontium-yttrium mixed carrier if Ra-228 analysis is requested.
- 11.4.5 Add 30 mL basic EDTA reagent; vortex thoroughly, and heat in a hot water bath (approximately 80°C) until precipitate dissolves. There should be no precipitate remaining in the tube.
  - 11.4.5.1 If insoluble solids remain in the tube after addition of EDTA, confirm that the pH is > 10.

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 9 of 13

- 11.4.5.2 If pH > 10, centrifuge and syringe filter the supernate into a clean, labeled 50 mL centrifuge tube. Discard insoluble residue.
- 11.4.6 Add 1 mL ammonium sulfate (200mg/mL).
- 11.4.7 Add 2 mL of 17.4M acetic acid or until barium sulfate precipitates and vortex thoroughly.
- 11.4.8 Digest in a hot water bath until precipitate settles.
- 11.4.9 Centrifuge and discard supernate to acid waste..
- 11.4.10 Add 30 mL basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves.
- 11.4.11 Add 1 mL ammonium sulfate (200mg/mL).
- 11.4.12 Add 2 mL of 17.4M acetic acid or until barium sulfate precipitates and vortex thoroughly.
- 11.4.13 Record the date and time of last barium sulfate precipitation (T1). This is the beginning of the <sup>228</sup>Ac in-growth time.
- 11.4.14 Digest in a hot water bath until precipitate settles.
- 11.4.15 Centrifuge and discard supernate to acid waste..
- 11.4.16 Add 20 mL basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves.
- 11.4.17 Add 0.2 mL standardized yttrium carrier and 1 mL lead carrier (1.5 mg/mL).
  - 11.4.17.1 If any precipitate forms, dissolve it by adding a few drops of 10N NaOH.
  - 11.4.17.2 If radium-228 analysis is requested, cap the tube and allow it to age at least 36 hours.
  - 11.4.17.3 If radium-226 analysis is requested, cap the tube and allow it to age (in-growth period is 14 or 21 days, see sample log in sheet).
- 11.5 Lead Scavenge Clean-up
  - 11.5.1 Add 0.3 mL ammonium sulfide. Add 0.5 mL of 10N sodium hydroxide until lead sulfide precipitates, vortex then centrifuge.
  - 11.5.2 Add 1 mL lead carrier (1.5 mg/mL), 0.1 mL ammonium sulfide, and 0.1 mL of 10N sodium hydroxide.
  - 11.5.3 Centrifuge and filter supernate through 0.45 mm syringe filter into a clean labeled tube. Discard filter.
    - 11.5.3.1 The half life <sup>228</sup>Ac is very short. Check with count room staff on scheduling of the GFPC. **Do NOT proceed with the remaining steps of this procedure until authorized by the count room GFPC analysts or Radiochemistry manager.**
- 11.6 Out of In-growth
  - 11.6.1 Once yttrium hydroxide is precipitated, the analysis must be carried to completion to avoid excessive decay of <sup>228</sup>Ac.
  - 11.6.2 Add 5 mL 18N sodium hydroxide, stir well and digest in a hot water bath, 70-85°C, until yttrium hydroxide coagulates, usually about 10 minutes. Centrifuge for 10 minutes and carefully decant supernate into a clean, labeled 50 mL centrifuge tube. Save for barium yield determination, Step 11.8.1
  - 11.6.3 Record time (T2) of yttrium hydroxide precipitation; this is the end of the <sup>228</sup>Ac in-growth time and the beginning of <sup>228</sup>Ac decay time.
- 11.7 Yttrium Yield (for radium-228)
  - 11.7.1 Weigh a stainless steel cleaned planchet. Record weight in the LIMS spreadsheet.
    - 11.6.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.
  - 11.7.2 Dissolve the precipitate from step 11.5.2 in 2 mL 6M nitric acid. Vortex and add 5 mL water and precipitate yttrium hydroxide with 3 mL 10N sodium hydroxide. Heat and stir in a hot water bath until precipitate coagulates. Carefully centrifuge for 10 minutes. Carefully discard supernate to base waste. (do not dump gel-like pellet).

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 10 of 13

- 11.7.3 Dissolve precipitate with 1 mL 2M nitric acid. Vortex.
  - 11.7.3.1 If solution is still cloudy add 2M nitric acid drop wise until the solution clears.
- 11.7.4 Dilute to 5 mL with DI water and add 2 mL 5% ammonium oxalate. Centrifuge and discard supernate to acid waste.
- 11.7.5 Wash precipitate with 10 mL DI water.
- 11.7.6 Vortex, centrifuge and discard supernate to acid waste.
- 11.7.7 Plating
  - 11.7.7.1 To determine yttrium yield, quantitatively transfer the precipitate to the previously weighed stainless steel cleaned planchet using a minimal amount of water.
  - 11.7.7.2 Dry on a hot plate.
  - 11.7.7.3 Upon dryness, let cool in a desiccator for a minimum of 15 minutes and then weigh planchet.
  - 11.7.7.4 Record the weight in the LIMS worksheet to determine the chemical yield of the yttrium carrier solution.
- 11.8 Barium Yield (for radium-226)
  - 11.8.1 Weigh a stainless steel cleaned planchet. Record weight in the LIMS spreadsheet.
    - 11.8.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.
  - 11.8.2 To the supernate from Step 11.5.2, add 5 mL 16M nitric acid and 2 mL ammonium sulfate (200mg/mL),. Add 5 mL 17.4M acetic acid until barium sulfate precipitates. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate to acid waste.
  - 11.8.3 Add 30 mL basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves. Add a few drops 10N NaOH if precipitates does not readily dissolve.
  - 11.8.4 Add 1 mL ammonium sulfate (200 mg/mL). Add 2 mL 17.4M acetic acid until barium sulfate precipitates and vortex thoroughly.
  - 11.8.5 Record date and time (T3) of BaSO<sub>4</sub> precipitate in the LIMS data sheet.
  - 11.8.6 Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate to acid waste.
  - 11.8.7 Wash precipitate with 10 mL water. Vortex, centrifuge and discard supernate acid waste.
  - 11.8.8 Plating
    - 11.8.8.1 Transfer the precipitate to a pre-weighed stainless steel cleaned planchet with a minimal amount of water.
    - 11.8.8.2 Heat the planchet again using the hot plate, let cool in a desiccator for a minimum of 15 minutes and then weigh planchet.
    - 11.8.8.3 Record the final weight of the planchet in LIMS to determine the chemical recovery for the barium carrier solution.
- 11.9 Submit the planchets to the counting room for analysis.

## 12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. LCS % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.
- 12.2 There are no calculations pertaining to this sample preparation procedure.
- 12.3 Radium-226 and radium-228 by GFPC calculations are given in ST-QAM.

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 11 of 13

### **13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA**

- 13.1 Data assessment does not pertain to this sample preparation procedure.
- 13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

### **14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY**

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in LIMS
- 14.2 Demonstration of Capability
  - 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.
- 14.3 Training Qualification
  - 14.3.1 The manager/supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
  - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.
- 14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

### **15.0 VALIDATION**

- 15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

### **16.0 WASTE MANGEMENT AND POLLUTION PREVENTION**

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2 Waste Streams Produced by the Method
  - 16.2.1 The following waste streams are produced when this method is carried out.
    - 16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
    - 16.2.1.2 Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid Rad waste for disposal by the EH&S Coordinator.

### **17.0 REFERENCES**

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 12 of 13

- 17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0, Radium-228 in Drinking Water
- 17.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0, Alpha-Emitting Radium Isotopes in Drinking Water
- 17.3 SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Method 9315, Alpha Emitting Radium Isotopes
- 17.4 SW-846, "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Method 9320, Radium-228
- 17.5 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.
- 17.6 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revision
- 17.7 Associated SOPs:
  - 17.7.1 ST-PM-0002, Sample receipt and Chain of Custody
  - 17.7.2 ST-QA-0002, Standard and Reagent Preparation
  - 17.7.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
  - 17.7.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
  - 17.7.5 ST-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis
  - 17.7.6 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
  - 17.7.7 ST-RD-0403, Gas Flow Proportional Counting (GFPC) Analysis

## 18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

- 18.1 After initial precipitation, TestAmerica St. Louis decants the supernate after the precipitate has been allowed to settle for at least six hours, as opposed to the EPA Method 904 which requires filtration to isolate the precipitate.
- 18.2 At the point of in-growth of actinium-228, TestAmerica St. Louis utilizes either a 14 days or 21 day in-growth depending on the client's request for wastewater and 21 days for drinking water before finishing the procedure.
- 18.3 TestAmerica St. Louis counts the barium sulfate fraction (minus the Ra-224) by GFPC to report radium-226; an option documented in section 10.5 of the EPA Method 903.0.

## 19.0 CHANGES FROM PREVIOUS REVISION

- 19.1 Annual Review, No Changes.
- 19.2 Rev. 10:
  - 19.2.1 Updated section 11.4 to add carrier prior to adding EDTA.
  - 19.2.2 Moved 11.4.15, recording the date/time of the last barium sulfide precipitation to section 11.4.9.
- 19.3 Rev. 11:
  - 19.3.1 Updated soil LCS composition in section 9.3.4.
  - 19.3.2 Updated amount of standardized yttrium carrier used in section 11.4.17
  - 19.3.3 Updated amount of DI water for precipitate wash and removed repetition of wash and vortex steps in section 11.7.7 and 11.7.8.
- 19.4 Rev. 12:
  - 19.4.1 Grammatical corrections throughout
  - 19.4.2 RadCap/RadCapture changed to LIMS.
  - 19.4.3 Deleted stir thoroughly

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SOP No. ST-RC-0041, Rev. 18

Effective Date: 03/08/2019

Page No.: 13 of 13

- 19.4.4 Section 11.3.3 changed time from 30 minutes to 5-10 minutes.
- 19.4.5 Section 11.3.5 added: add 10 mL, and deleted add 0.25 mL more.
- 19.4.6 Section 11.4.5.2 added syringe filter supernate into clean labeled tube.
- 19.4.7 Section 11.4.7 changed aliquot to 3 mL of acetic acid.
- 19.4.8 Section 11.4.9 moved to after 11.4.13.
- 19.4.9 Section 11.4.13 changed aliquot of acetic acid to 3 mL.
- 19.4.10 Section 11.5.1 removed stir well after ammonium sulfide is added, and changed aliquot of sodium hydroxide to 0.5 mL.
- 19.4.11 Section 11.5.2 changed aliquot of sodium hydroxide to 0.1 mL.
- 19.4.12 Section 11.8.2 deleted stir well after each addition and changed aliquot to 7 mL acetic acid.
- 19.4.13 Section 11.8.4 changed aliquot of acetic acid to 3 mL.
- 19.4.14 Updated section 15
- 19.5 Revision 13: (07/31/2014)
  - 19.5.1 Section 7.8 added the name of the reagent, BaSO<sub>4</sub>
  - 19.5.2 Section 7.8.2 changed 16 to 20 mg/ml, Ba per certificate
  - 19.5.3 Section 7.17 made correction for the concentration, replaced 9 with 10 mg/ml per method.
  - 19.5.4 Section 7.19 added concentration, 5 mg/ml
  - 19.5.5 Section 7.2 Corrected concentration from 9 to 2.5 mg/ml.
- 19.6 Revision 14: (08/11/2015)
  - 19.6.1 Updated section 7.0
  - 19.6.2 Updated section 11.0
- 19.7 Revision 15: (06/30/2016)
  - 19.7.1 Updated section 11.0
  - 19.7.2 section 11.4.5, 11.4.10, and 11.8.3, the amount of EDTA was increased from 20 mL to 30 mL.
  - 19.7.3 section 11.4.6, 11.4.7, 11.4.11, 11.4.12, and 11.8.4, the amount of ammonium sulfate and acetic acid added was changed.
  - 19.7.4 Section 11.6.2 the amount of sodium hydroxide and the time was updated. Recording the time of the yttrium hydroxide precipitation was moved to this step
- 19.8 Revision 16 (06/30/17)
  - 19.8.1 Updated section 5.0- changed from MSDS to SDS
  - 19.8.2 Updated section 7.0- clarified non-critical reagents, changed concentrations of reagents, removed "from Milli Q Unit"
  - 19.8.3 Updated section 11.0- updated procedure- centrifuge times, where to dump supernate, added information about T-times
- 19.9 Revision 17 (05/17/2018)
  - 19.9.1 Technical Review: S.Bernsen /QA Review M Ward
  - 19.9.2 Updated section 6.6- added additional syringe volumes, 50 mL and 60 mL; removed 0.45mm filter
- 19.10 Revision 18 (03/08/2019)
  - 19.10.1 Technical Review: C. Mazariegos/QA M Ward
  - 19.10.2 Updated section 11.0
    - 19.10.2.1 Section 11.3.1 changed "red color" to "pink endpoint" and added "for soils skip to 11.3.4."
    - 19.10.2.2 Section 11.3.2-3 moved spiking and tracing prior to adding any reagents that could cause a precipitation.
    - 19.10.2.3 Section 11.3.6 added "slowly add" and "If no yellow endpoint is visible, insure pH is > 6.5."
    - 19.10.2.4 Section 11.3.7 updated to "stir then heat until close to boiling for about 30 minutes."
    - 19.10.2.5 Section 11.3.8 changed "red color" to "pink endpoint".
    - 19.10.2.6 Section 11.3.9 added "until a barium sulfate precipitate forms."
- 19.11 Added Eurofins logo and updated copyright information (4/19/2019)

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## **Appendix F**

# **ISO-PACIFIC Soil Sorting Operations Work Plan**

# **FINAL SOIL SORTING OPERATIONS WORK PLAN**

**HUNTERS POINT NAVAL SHIPYARD  
SAN FRANCISCO, CALIFORNIA**

**January 2020**

**Prepared for:**



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# **FINAL SOIL SORTING OPERATIONS WORK PLAN**

HUNTERS POINT NAVAL SHIPYARD  
SAN FRANCISCO, CALIFORNIA

January 2020



02/17/20

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Mike Dillon  
Technical Lead

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Date



02/17/20

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Lori Dillon  
President

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Date

## Table of Contents

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1.0	INTRODUCTION .....	1
1.1	S3 Tasks and Actions .....	1
1.2	Pre-Mobilization Activities .....	1
1.3	S3 Personnel and Staff Training Requirements .....	2
1.4	S3 Mobilization / Decontamination / Demobilization .....	2
1.5	Ancillary Support Equipment .....	3
1.6	S3 General Process Overview .....	3
1.6.1	Soil Pre-Conditioning .....	3
1.6.2	Soil Distribution .....	4
1.6.3	Final Shaping of Soil for Scanning .....	4
1.6.4	The Radiation Detection System .....	4
1.6.5	Radioassay of the Soil .....	5
1.6.6	The Diversion Sequence in Detail .....	6
1.6.7	Database Functions .....	7
1.6.8	The Human-Machine Interface .....	7
1.7	Inclement Weather Considerations .....	7
2.0	S3 DETECTOR ARRAY INSTRUMENTATION .....	8
2.1	Detectors and Array Geometry .....	8
2.2	Detector Shielding Structure .....	8
3.0	S3 TECHNICAL BASIS .....	10
3.1	Feed Soil Sizing and Soil Stream Parameters .....	10
3.2	Belt Speed and Soil Depth .....	10
3.3	Soil Density .....	12
3.4	Energy Calibrations .....	13
3.5	Traceable Calibration Sources .....	14
3.6	ROI Settings .....	14
3.7	Background .....	15
3.8	S3 Efficiency Determinations .....	16
3.9	Secular Equilibrium .....	16
3.10	S3 Activity Calculations .....	18
3.11	S3 Minimum Detectable Concentrations .....	18
3.12	S3 Alarms .....	19
3.13	Alarm Summary .....	19

4.0	PRE-OPERATIONAL TESTING AND OPERATIONAL CHECKS .....	21
4.1	<sup>226</sup> Ra Object Detection Verification .....	21
4.2	Distributed Contamination Calibration Verification .....	22
4.3	Daily S3 Function and Source Response Checks .....	22
4.4	S3 Control Charts.....	23
5.0	LLRO RETRIEVAL AND SAMPLING EVOLUTIONS .....	26
5.1	Object Retrieval from the Assay Belt.....	26
5.2	Strontium Marker Detection.....	26
5.3	Timed Soil Sampling Activities .....	27
5.4	Transfer of Excavated Soil to the S3 for Processing .....	27
5.5	Above Criteria Sampling Evolutions .....	28
6.0	DATA COLLECTION AND REPORTING .....	29
6.1	S3 Data Collection .....	29
6.2	Human – Machine Interface .....	31
6.3	Documentation Quality Standards.....	32
6.4	Record Retention .....	33
7.0	REFERENCES .....	34

## List of Figures

---

Figure 1-1	Organization Chart and Role and Responsibilities
Figure 1-2	S3 Operations in the Rain
Figure 1-3	View from the S3 Control Room
Figure 1-4	Inclement Weather and Extreme Cold Conditions
Figure 2-1	NAIS Centroid Variation for Temperature Range 10-30°C
Figure 2-2	Mirion Canberra Model NAIS 3x5x16 Temperature Stabilized Sodium Iodide Scintillation Detector
Figure 3-1	Gamma Transmission as a Function of Energy and Soil Depth (HydroGeologic, 2012)
Figure 3-2	An S3 Energy Calibration Curve Developed by a Genie-2000 Library File
Figure 3-3	Control Chart for S3 Detector 1, Initial Calibration Data for Empty Belt Background
Figure 3-4	An Example of a Belt Sampling Template Used in On-Conveyor Soil Density Determinations (Photo Is Fig. 1 from the ASTM D75/D75M Document)
Figure 4-1	The S3's Sampling Point on the Assay Belt
Figure 4-2	The S3's Bremsstrahlung Spectrum Resulting from 90Sr Beta Emission in Soil and Interaction with the System's Soil and High-Z Materials
Figure 4-3	Plan View of a Typical S3 Footprint Orientation
Figure 5-1	Example of the S3's Batch Chart Output
Figure 5-2	A Human-Machine Interface Page in the S3 Software Highlighting the Software's Display of a Found Source on the Belt of the S3, Shown In Red

## List of Tables

---

Table 1-1	S3 NaI Detector Regions of Interest
Table 3-1	Instrument % Efficiency Values for Specified Regions of Interest Under a Range of S3 Operating Parameters
Table 3-2	Minimum Detectable Concentrations (pCi/g) for Diffuse $^{226}\text{Ra}$ and $^{137}\text{Cs}$
Table 3-3	Minimum Detectable Activity ( $\mu\text{Ci}$ ) for a $^{226}\text{Ra}$ LLRO
Table 3-4	Equilibrium-Modified Instrument % Efficiency Values and Minimum Detectable Concentrations for Diffuse $^{226}\text{Ra}$ (ROI 1) Under a Range of Operating Parameters*
Table 3-5	Equilibrium-Modified Instrument % Efficiency Values and Minimum Detectable Concentrations for Diffuse $^{232}\text{Th}$ ( $^{228}\text{Ac}$ , ROI 5) Under a Range of Operating Parameters*
Table 3-6	Equilibrium-Modified Instrument % Efficiency Values and Minimum Detectable Concentrations for Diffuse $^{232}\text{Th}$ ( $^{208}\text{Tl}$ , ROI 6) Under a Range of Operating Parameters*

## Acronyms and Abbreviations

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<sup>90</sup> Sr	strontium-90
<sup>137</sup> Cs	cesium-137
<sup>222</sup> Rn	radon-222
<sup>226</sup> Ra	radium-226
<sup>235</sup> U	uranium-235
<sup>232</sup> Th	thorium-232
<sup>228</sup> Ac	actinium-228
<sup>208</sup> Tl	thallium-208
<sup>239</sup> Pu	plutonium-239
<sup>152</sup> Eu	europium-152
CRO	Control Room Operator
ESU	excavation soil unit
HAZWOPER	Hazardous Waste Operations and Emergency Response
HPNS	Hunters Point Naval Shipyard
ISO	ISO-Pacific Remediation Technologies, Inc.
ISOCs	In-Situ Object Counting System
keV	kilo electron volt
LLRO	low-level radiological object
LLRW	low-level radioactive waste
MCNP	Monte Carlo N-Particle code
MDA	minimum detectable activity
MDC	minimum detectable concentration
NaI	sodium iodide
NAIS	stabilized sodium iodide detector
Navy	U.S. Department of the Navy
NIST	National Institute of Standards and Technology
NVLAP	National Voluntary Laboratory Accreditation Program
OSHA	Occupational Health and Safety Administration
PRSO	Project Radiation Safety Officer
RCT	radiological control technician
RG	remediation goal
ROC	radionuclide of concern
ROI	region of interest
S3	ISO-Pacific Soil Sorting System
SAP	sampling and analysis plan
SCADA	Supervisory Control and Data Acquisition
SFU	sidewall floor unit
SSOP	Soil Sorting Operations Work Plan
VFD	variable frequency drive



## 1.0 INTRODUCTION

---

### 1.1 S3 Tasks and Actions

This Soil Sorting Operations Work Plan (SSOP) presents the specific tasks and actions needed for screening, scanning, and diverting soil for potential radiological contamination at Hunters Point Naval Shipyard (HPNS), San Francisco, California. This SSOP was prepared to apply to Aptim Federal Services, LLC (APTIM) projects at HPNS. Soil sampling of screened soil will be performed as discussed in project-specific work plans and sampling and analysis plans (SAPs). Radionuclides of concern (ROCs) vary by project.

The objective of this SSOP is to implement the following work elements:

- Operation of the S3 in accordance with standard operating procedures to produce soil sorting characterization reports customizable to project requirements with source peak radioactivity reports for detected Low-Level Radioactive Objects (LLROs) and diffuse soil contamination.
- The ROC to be screened using the S3 include radium-226 ( $^{226}\text{Ra}$ ) and thorium-232 ( $^{232}\text{Th}$ ) progeny, and cesium-137 ( $^{137}\text{Cs}$ ). Compliance with  $^{137}\text{Cs}$ , strontium-90 ( $^{90}\text{Sr}$ ), plutonium-239 ( $^{239}\text{Pu}$ ), and uranium-235 ( $^{235}\text{U}$ ) remediation goals (RGs) will be based on systematic soil sample results in accordance with project-specific work plans and SAPs. Note that ROCs at HPNS vary by site and/or parcel.
- To accurately characterize HPNS soil, the soil sorter will scan 100% of the soil stream to identify and divert soils and LLROs with radiological activities that equal or exceed established criteria for specified ROCs or their gamma-emitting surrogates.
- Systematic soil sampling will be conducted in accordance with project-specific work plans and SAPs to demonstrate that the screened below criteria stockpile contains ROC concentrations below RGs. When combined with the scanning step using the soil sorter, these combined processes ensure that the primary objective of the survey is achieved (i.e., residual radioactivity above the RG is removed for disposal as radiologically-impacted waste).

### 1.2 Pre-Mobilization Activities

At least two weeks prior to mobilization, APTIM will notify the appropriate U.S. Department of the Navy (Navy) personnel, including the Navy Remedial Project Manager, Resident Officer in Charge of Construction, and Caretaker Site Office representative regarding the planned schedule for S3 arrival. The Navy will notify the agencies. Upon receipt of the appropriate records and authorizations, field personnel, temporary facilities, and the S3 plant will be mobilized to the site.

Temporary facilities needed to support the S3 operations may include one or more secure storage (Conex) boxes for short- and long-term storage of materials, a portable generator, mechanical screening equipment, and soil conveyors.

### **1.3 S3 Personnel and Staff Training Requirements**

The S3 is operated by two ISO-Pacific Remediation Technologies, Inc. (ISO) personnel: a control room operator (CRO) and a lead plant operator. A radiological control technician (RCT) is also trained to support S3 operations and collect soil samples. An organization chart including roles and responsibilities is provided as Figure 1-1.

Non-site-specific training for S3 field personnel will be performed before mobilization to the extent practical. Medical examinations, medical monitoring, and medical training will be conducted in accordance with the requirements in the HPNS project-specific work plans. In addition to health and safety-related training, other training may be required as necessary, including but not limited to the following:

- Fall Protection (for personnel working at heights greater than 5 feet)
- Equipment Safety as required for ISO workers (screen plant, generator set, stacker conveyors)
- On-site S3 Safety Training for Contractor Workers (laborers, operators, RCTs and samplers)
- On-site Radiological Worker Training and Qualification

Other Health and Safety Training may include, but is not limited to, the following:

- Occupational Safety and Health Administration (OSHA) 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training
- OSHA 8-hour HAZWOPER refresher training
- OSHA-required On-The-Job training
- Site- and task-specific activity hazard analysis training

### **1.4 S3 Mobilization / Decontamination / Demobilization**

Mobilization activities will include movement of personnel, equipment, and materials to the site, and orientation and training of field personnel. Site-specific training will include radiological safety awareness. Personnel working at HPNS will acquire badges by the Office of Community Investment and Infrastructure during mobilization.

The S3 plant system and associated equipment will be surveyed by APTIM RCTs prior to initial use and prior to demobilization. Decontamination, if required, shall be performed as described in the respective project-specific work plan. Decontamination wastes will be properly containerized and characterized according to the project-specific waste management plan. If the wastes are determined to be low-level radioactive waste, the waste will be transferred by APTIM to the designated Navy low-level radioactive waste broker.

## **1.5 Ancillary Support Equipment**

The following ancillary support equipment will be used in conjunction with operation of the S3:

- A mechanical screen to provide minus 2" particle size feed material.
- Heavy earthmoving equipment to feed the screen with raw soil.
- Stacking conveyors, positioned under the reversing diversion belt to move soil away from both the Diverted Material and the below criteria material sides of the plant. Additional stackers maybe used if needed.
- A portable generator capable of providing 480-volt, 30 amp, three-phase electrical service.

## **1.6 S3 General Process Overview**

This section provides a beginning-to-end description of the soil sorting operation and decision-making process. Dust management is discussed in the project-specific dust management plan. A more detailed technical basis discussion of the following sections is provided in Section 3.0.

### **1.6.1 Soil Pre-Conditioning**

The S3 uses a screen plant to properly size the soil for processing, and the first component that HPNS soil will encounter is this screen plant. The screen is used to break up cohesive soils and remove oversize material consisting of rock, debris, and other materials greater in diameter than the S3's process soil layer depth. The specific type of screen plant used will produce soil that can be deposited in the system in a loose and flowable condition. Tracked screen plants are preferred so that they can be moved easily, as spilled soil buildup occurs under the unit and this spillage will be cleared away periodically and processed through the S3.

Screen mesh sizing is based on the layer thickness desired, soil moisture conditions, rock content, and economic factors. Screening does result in some oversize material, and the

fate of the oversize fraction must be considered. Oversized debris will be radiologically screened and processed as described in project-specific work plans.

### **1.6.2 Soil Distribution**

Screened soil enters the S3 sorting system via the surge bin and is deposited into this bin by a conveyor attached to the screen plant. The bin's function is to mitigate uneven flows from the screen plant and to distribute soil evenly on the S3's assay conveyor belt. It does this by use of an adjustable height mechanism to position a flat screed to set the initial thickness of the soil layer on the assay belt. When the assay belt moves at a constant pre-selected speed, soil exits the bin and the integral screed shapes the soil into a thin, flat, wide stream. The actual soil layer thickness is set depending upon the isotopes of concern and corresponding sensitivity data quality objectives.

An S3 operator stationed near the surge bin visually observes the produced soil stream and will remove occasional debris such as tramp metal, string, weeds, roots, and other detritus that may pass through the screen plant and disturb the soil layer geometry.

### **1.6.3 Final Shaping of Soil for Scanning**

The S3's Secondary Screed, downstream of the surge bin, acts as a final re-shaper of the soil stream before it passes under the S3's detector array. As the initial stream produced by the surge bin is slightly thicker and narrower than the Secondary Screed, the screed strikes the soil to the required thickness, and by virtue of its plow shape, moves struck soil outward to the edges of the conveyor belt, widening the soil stream. Thus, adjustments to the initial surge bin depth settings, and the subsequent response by the Secondary Screed will define the ultimate thickness and width geometry of the S3's soil stream.

### **1.6.4 The Radiation Detection System**

The S3's detector array is immediately downstream of the Secondary Screed and monitors the radioactive energy emissions from the contaminant material in the soil. Eight large volume sodium iodide (NaI) scintillator detectors are used. Scintillating crystal detectors are preferred for performing scanning surveys for several reasons: they are sensitive, rugged, and require no detector cooling.

When a gamma ray enters a scintillation detector crystal, electronic interactions inside the crystal can cause light to be emitted. The amount of light emitted is proportional to the energy of the gamma ray absorbed. These light flashes are detected by a photomultiplier tube (PMT) coupled to the detector. The PMT converts light flashes to electrical charges, which in turn are measured by a pulse height analyzer.

The detector array is equipped with devices called multichannel pulse height analyzers (MCA), which perform the function of determining the number of pulses of a given height

detected over a period of time. The system's computer software processes the digital output of the MCA in count rates per second.

Each MCA will define a number of spectrographic regions of interest (ROI). Active ROIs will alarm if an RG threshold is exceeded, while others are used strictly for data collection purposes. Table 1-1 lists the separate ROIs along with each ROI's purpose, relationship to an ROC and, the associated RG.

In general, the larger the NaI crystal, the more gamma rays from a given source will be converted to detector counts. The thickness of the crystal also affects the efficiency of the absorption of gamma rays of various energies, and thicker detector crystals are very useful for the detection of uranium- and thorium-series isotopes that have prominent high-energy gamma rays. Photon-emitting daughter products associated with uranium, thorium, and radium exist in various states of equilibrium with their parent nuclide and can be used, in certain circumstances, as effective surrogates for isotopes that otherwise emit primarily difficult-to-detect alpha radiation. The MCA and NaI detector combination serve to efficiently detect and bin a wide-range of photon energies into ROIs associated with specific ROCs with sufficient resolution to discriminate energies caused simultaneously by ROCs or their photon-emitting daughter progeny.

Use of lead shielding in the S3's detector box and the surrounding structure maintains a low background radiation contribution to the process. Adjusters built into the detector box will raise or lower the detector array to set the pre-determined final height of the detector faces above the soil stream.

### **1.6.5 Radioassay of the Soil**

As soil on the assay belt leaves the Secondary Screed and passes under the array of radiation detectors, the S3's proprietary software algorithms continually monitor the data feeds from each detector's MCA. The MCA maintains two clocks during acquisition: real-time and live time. Whenever a count rate is reported or used in a calculation, it is always expressed as counts/live time. Dead time (DT) corrections are applied in the S3 software early in the calculation and analysis chain on a per second basis. The uncertainty on the DT correction is minimal and the overall uncertainty will be dominated by the 'sqrt(N)' counting statistics. This information is streamed at one second intervals to the system's software for the activity calculation and logging and to enable the software to display data from each instrument. Refer to Section 3 for additional details on mathematical processes to transform NaI scanning system output into results suitable for comparison to volumetric RGs.

The entire radioassay process is automatically controlled by the system's Supervisory Control and Data Acquisition (SCADA) based software. SCADA is a control system architecture that uses computers, networked data communications, and graphical user interfaces for high-level process supervisory management. If this system determines that a soil segment under scrutiny exceeds one of the pre-defined site RGs, it then flags those

portions of the soil for mechanical separation from the surrounding below criteria soil and will automatically direct the diversion of that soil to the Diverted Material pathway.

### **1.6.6 The Diversion Sequence in Detail**

Since the SCADA system is programmed with all the geometric constants of the conveyor systems, including the belt speeds of both the assay belt and the reversible diversion conveyor, and the physical location of the radiation detectors in relation to the head pulley of the assay belt and to each other, the software 'knows' exactly where the discovered nodes of contamination are on the conveyor belts at any one time, and the physical extent of the contamination.

As the S3's algorithms monitor the location of the soil on the moving belt, the diversion sequence will automatically sort the soil in one of two categories; material screened as non-LLRW (below criteria material that are less than applicable RGs ( $<RG$ ), and material screened as potential LLRW (Diverted Material suspected of containing LLRO(s) or diffuse contamination potentially exceeding an associated RG ( $>RG$ ). Mechanical diversion of soils is achieved by a control signal to the reversing diversion belt, which is located transverse to the discharge point of the assay belt.

As soils pass under the detector array and are monitored as  $<RG$ , the main assay belt discharges onto a reversible diversion belt that will discharge that soil to the  $<RG$  stockpile.

If the S3 software detects an LLRO or soil activity above an RG-based count rate threshold alarm setpoint (i.e., a Diversion Event), the system will begin tracking the length of soil on the assay belt that exceeds an RG. The assay belt comes to a complete stop before the suspect soils reach the drop point to the reversible diversion belt. The diversion belt will continue to remove "clean" soil that has already dropped onto the diversion belt to the below criteria material (i.e., "clean") pile. Once "clean" soils have been removed from the diversion belt to the "clean" pile, it will reverse directions. Then, the assay belt restarts. As the suspect objects/soils drop from the assay belt, the diversion belt reverses direction and sends the suspect materials to the Diverted Material bin for further assessment or management and disposal as radiological waste, as appropriate. As the last of the suspect material drops to the diversion belt, the main assay belt comes to a complete stop until the diversion belt has sent all suspect soils to the Diverted Material bin. Belt directions and scanning operations then return to normal and the Diversion Event is recorded in S3 histories.

If the system detects another radiological object or block of diffuse soil contamination arriving within three seconds of the end of the areal extent of the first block of discovered material, it will flag the assay belt to ignore the small segment of 'clean' soil in between the two, and will continue to run until both blocks of suspect soil have been deposited on the diversion belt for transfer to the Diverted Material bin. Overall, this process ensures that suspect materials are reliably diverted with minimal unnecessary diversion of  $<RG$

soils; as little as 1.5 cubic feet of soil may be isolated as a result of a single Diversion Event.

Confirmatory sampling of both Diverted Material and below criteria material is conducted as described in the project-specific work plans and SAPs.

Both the main assay belt conveyor and the reversing diversion conveyor are equipped with tensioned scraper systems which create a constant knife-edge of polymeric material or metal on the belt surface at the head pulley location or underneath the belt. The knife edges scrape soil particles, mud, water, ice, or snow buildup off the belting. Both conveyor belts are constructed of stabilized rubber polymers which resist cross-contamination, static electricity effects, and prevent or retard degradation of the polymers by heat, light, and atmospheric exposure.

#### **1.6.7 Database Functions**

The S3's software accommodates overlapping alarm signals from the various ROIs. The algorithm tracks the areal extent discovered using the time information in all the activity databases and schedules the resulting diversion times accordingly. All diversion events are logged by software historians, captured onto a database feature of the SCADA system to archive and retrieve high-fidelity time series data.

#### **1.6.8 The Human-Machine Interface**

All data collection systems are directly wired into the onboard Control Room. Here, the system uses an industrial computer running the SCADA program to control all processes. The SCADA system automatically gathers and monitors the entire system's digital information, logs that information to the computer and other peripheral drives, and carries out all necessary analysis and machine control based on the programming parameters assigned to it. The system is programmed to display the information in a logical and organized fashion on a software-developed human/machine interface screen.

### **1.7 Inclement Weather Considerations**

The sorter system has historically processed highly saturated clay soils inundated by rainstorms and snowfall in the past without issue. A 2013 sorting project in the Midwest was conducted in severe inclement weather and sub-zero temperatures, causing repeated heavy equipment failures and significant system downtime due to unexpected generator shutdowns due to cold-induced gelling of the local diesel fuel supply. Despite the adverse weather conditions, the S3 reported a 100% availability rate. See Figures 1-2 through 1-5 for images of these conditions. Dust control and stormwater management are described in project-specific work plans.

## 2.0 S3 DETECTOR ARRAY INSTRUMENTATION

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### 2.1 Detectors and Array Geometry

The S3 uses eight temperature and energy gain stabilized NaI detectors (NAIS) in its array. The NAIS has a crystal thickness of 3", a width of 5", and is 16" in length (3x5x16). The NAIS detector has a drift of less than 2% in peak centroid at 662 keV over a temperature range of -4° to 122°F. The detector achieves temperature stabilization with internal stabilization electronics, and each detector in the array is connected to an Osprey™ digital tube-based multichannel analyzer (MCA). The typical resolution of the NAIS detector is 7.5% at 662 keV.

Figure 2-1 compares the measured  $^{137}\text{Cs}$  peak centroid measured by a standard 3x5x16 detector with the NAIS 3x5x16 detector over a temperature range of 10-30 °C. A photograph of an NAIS detector is shown in Figure 2-2.

The S3's detector array geometry has been designed with Mirion Canberra's *In-Situ* Object Counting System (ISOCS) and verified with Los Alamos National Laboratory's Monte Carlo N-Particle code (MCNP). Two categories of proposed geometries were investigated using the S3's array of large volume NaI detectors. The first geometry assumed the radionuclides of interest were homogeneously mixed in the soil matrix, while the second geometry modeled the source as discrete point sources completely buried underneath the soil matrix and passing directly between two adjacent detectors as a bounding worst-case. Using both scenarios, the number and placement of detectors were fine tuned to develop the geometry for the detector array.

The detector array is adjusted vertically using linked five-ton screw jacks mounted to the detector box. This allows the optimum soil surface to detector array distance to be set for the best field of view. The air gap distance from the face of the detectors to the top soil surface is set at three quarters of an inch and was determined *a priori* via MCNP modeling.

### 2.2 Detector Shielding Structure

The array of eight NAIS detectors are suspended inside a 5/8-inch thick steel detector box lined with 2 inches of poured lead in the top and sidewalls. A 1/4-inch bottomless steel shell completes the interior of the box. An insulated thin titanium sheet (0.016 inches thick) acts as a 'window' on the bottom of the box and protects the detector faces from the outside environment.

Below the detector box and under the assay conveyor's belting is a 1/2-inch thick steel plate supporting a 2-inch thick layer of interlocking lead bricks that creates a wide 'shadow shield' under the detector array. This layer virtually eliminates shine from the soil and other materials below the S3 and from nearby soil piles. A deeply chorded I-beam spans the width of the assay belt just upstream of the detector box has 1-inch interlocking lead



bricks concealed in both sides of the I-beam's web structure and is sealed on both sides with 1/4-inch thick sheet steel. This heavy 'side shield' reduces photon flux from the S3's surge bin, which is upstream of the detector box and may contain collected contaminated soils that might otherwise have a negative impact on the background environment of the detectors. In total, there is approximately 4 tons of lead and over 1.5 tons of steel shielding the detector array.

### 3.0 S3 TECHNICAL BASIS

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#### 3.1 Feed Soil Sizing and Soil Stream Parameters

The S3's feed soil must be screened to minus 2 inches to conform with the system's soil layer depth requirements prior to sorting. Soil pre-conditioning can be achieved by the use of screen plant systems that break up cohesive soils and remove oversize material (debris and rock with a diameter larger than the S3's soil depth on the assay belt). Specific screening machines are used with different soil types to provide a product that is loose and flowable. The screened soil is sent to the S3's surge bin by conveyor, which deposits the soil into the bin and onto the moving assay belt. The surge bin uses an adjustable screed to create the desired soil stream thickness and stream width on the belt.

Debris (larger than 2 inches) will be mechanically screened and segregated in accordance with current HPNS protocol to not interfere with radiological soil screening. Segregated debris will not be screened through the S3 and will be radiologically surveyed in accordance with project-specific work plans.

Gamma transmission through soil depends on both the gamma energy and the soil thickness and must be considered when defining a soil thickness. The National Institute of Standards and Technology (NIST) describes the gamma transmission variable (the percentage of gamma radiation which penetrates the soil) as a function of gamma energy and selected soil thicknesses<sup>1</sup>.

Larger belt soil thicknesses have a great effect on the transmission of gamma energies particularly when the source is attenuated by otherwise clean soils on the belt. Figure 3-1 illustrates the source transmission relationship photon energies between 0.06 and 3.0 mega electron volt at varying depths. ISO determined a soil thickness between one and three inches is optimal from a scanning efficiency perspective while permitting sufficient photon transmission to meet key ROC minimum detectable activity/concentration objectives.

#### 3.2 Belt Speed and Soil Depth

During the design of the detector array, an MCNP and ISOCS modeling study performed by Mirion Canberra first assessed the S3's ability to reliably detect and divert a discrete <sup>226</sup>Ra object with an activity of 0.02  $\mu$ Ci in a conservative belt location (i.e., between two detectors and resting on the conveyor belt covered by otherwise uncontaminated soil). The Mirion Canberra modeling study was based on an object with 0.02  $\mu$ Ci activity. For HPNS, the target LLRO activity is 0.087  $\mu$ Ci. This target MDA is based on consistency with previously approved radiological instruments currently in use at HPNS (CB&I Federal Services, LLC, 2015 [14]). Based on LLROs discovered at HPNS from 2016 to present, the smallest LLRO is 0.76  $\mu$ Ci <sup>226</sup>Ra and the average LLRO is 22  $\mu$ Ci <sup>226</sup>Ra. Therefore, a target LLRO MDA of 0.087  $\mu$ Ci is conservative for HPNS and is consistent with LLRO MDAs used at other Navy San Francisco Bay Area sites.

The Mirion Canberra modeling study also modeled efficiency values and calculated MDCs for gamma-emitting ROCs over conveyor belt speeds ranging from two to seven inches per second and soil thicknesses covering the object ranging from two to three inches. The process was then repeated assuming an RG was homogeneously distributed in a volume of soil passing under the array on the S3 conveyor belt; the isotopes modeled volumetrically in the study included  $^{137}\text{Cs}$ ,  $^{226}\text{Ra}$  (with daughters), and  $^{232}\text{Th}$  (with daughters). Belt speed and soil thickness will be limited to the extent that the actual MDC for a site-specific ROC ( $^{226}\text{Ra}$ ,  $^{137}\text{Cs}$ , or  $^{232}\text{Th}$ , as identified in the project-specific work plan) must be less than the corresponding RG.

The S3's soil stream is designed so that it does not touch the immovable conveyor skirting on either side of the assay conveyor, as that action might dislodge a LLRO from its assayed position on the conveyor belt. Thus, the S3's soil width parameter is defined as the distance from skirting to skirting less a certain dimension in inches on each side. Those dimensions are empirically derived while in the field from the soil depth and a soil's angle of repose on the belt but are typically set at two inches.

Variable Frequency Drive (VFDs) inverters installed on the S3 control the system's conveyors speed of rotation. These sensitive electronic modules are directly mounted on the electrical motors driving the gearboxes that rotate the head pulleys of the S3's conveyors. The VFD speed settings are automatically maintained by sensor-less current vector control and deliver a constant speed output irrespective of torque and electrical load factors on the motor.

Setting the inverter speed variable is performed with NORDCON, an onboard PC program used by the S3 operators that enables parameterization and overview of all S3 VFDs produced by Getriebebau Nord. The following belt related operating parameters are adjustable by the ISO-Technical Lead and will be documented when each adjustment is made. The Navy and agencies will be notified of changes to operating parameters:

- **SOIL DEPTH** is the thickness of the soil layer on the sorter belt. For HPNS, this parameter is initially set at two inches. Study modeling has determined the depth may be increased up to three inches and will meet MDC sensitivity objectives including efficiency compensation for disequilibrium conditions in  $^{226}\text{Ra}$ . However, if the thickness is increased above two inches, MDCs shall be recalculated and verified, in the field in real-time, by ISO to remain below applicable RGs using observed background and disequilibrium conditions.
- **SOIL WIDTH** is the width of the soil layer on the S3's assay belt. For HPNS, this parameter is set to no more than 4.75 feet to optimize the source geometry of soils passing beneath the detectors closest to the array edges.
- **BELT SPEED** is the constant velocity of the sorter belt. For HPNS, this parameter is initially set at six inches per second.

### 3.3 Soil Density

*Ex-situ* soil density will vary to some degree at HPNS due to handling, weather, physical screening, and other factors. Efficiencies and MDCs were calculated for conveyor soil densities ranging from 0.9 to 1.6 g/cc with resulting diffuse soil MDCs less than corresponding RGs. ISO will conduct initial and ongoing density tests during operations whenever the S3 operators note a significant change in materials, or change in physical characteristics.

The S3's soil density variable is defined by the actual weight of the soil on the assay belt, as delivered by the S3's surge bin, not the *in-situ* density, nor the density of soil sampled from a pile. This variable is determined by weighing a known volume of site soil type taken from the sorter belt during calibration operations and its density in grams per cubic centimeter is calculated. The density variable is determined in the field. The samples used in the density calculation are collected following the guidance in ASTM standards D7204-15, Standard Practice for Sampling Waste Streams on Conveyors and ASTM D75/D75M – 19, Standard Practice for Sampling of Aggregates.

The scale used for weighing the soil samples for S3 density determination is certified by the manufacturer to meet the accuracy standards set by the International Organization for Standardization (ISO). The accuracy of the scale is verified with a National Voluntary Laboratory Accreditation Program (NVLAP) certified test weight. The NVLAP certification meets ISO 17025 and ANSI (American National Standards Institute)/NCSL (National Conference of Standards Laboratories) Z-540-1 standards. The accuracy of the scale will be verified with the certified test weight prior to every use. The pre-use verification will be documented in the S3 daily operations log. Unsatisfactory verification tests will be reported to the ISO Field Lead, and the scale will be tagged out-of-service.

Section 7.3 of ASTM D7204-15 – *Sampling Open Belt and Tray System* describes the use of bulkhead templates on the conveyor to establish the sample gathering boundaries. Section 5.3.2 of ASTM D75/D75M -19 - *Sampling from the conveyor belt* provides a more detailed method for obtaining a representative sample from a conveyor belt. This method describes the use of templates for collecting at least three approximately equal increments, selected at random, from the conveyor belt being sampled.

To gather the samples used for density calculations, the S3 operators stop the conveyor belt while the sample increments are being obtained. The operators insert the fixed templates into the soil stream, the shape of which conforms to the flat shape of the S3's assay belt, spaced apart such that the material contained between them will yield an increment of the required size (see Figure 3.4 for an example of the template construction). The operators then carefully scoop all gross material between the templates into a suitable container, and then collect the remaining fines on the belt with a brush and pan and add that material to the container.

The mass of the sample collected is then divided by the sample volume to determine the density (Equation 1). The volume used in the calculation is the length and width of the

soil stream captured inside the sampling templates, and the height is the soil depth (not the height of the templates) developed by the Secondary Screed, initially set at 2 inches.

#### Equation 1:

$$\text{Soil density (g/cc)} = \text{Soil mass (g)} / \text{Soil volume (cc)}$$

In lieu of combining the samples and then quartering or splitting them, the S3 operators will weigh and calculate the density for each sample taken, and then determine the average density value from those values.

- **SOIL DENSITY** is a moist soil density measurement and will typically be near 1.6 g/cc, depending on the project's soil types and moisture content. Field measurements will determine the exact values to be used at HPNS.

Once determined, the soil density is entered into the S3 software and used as a variable for activity calculation purposes. This density value is captured in a S3 parameter record historian and is reported in the daily run data batch report. Depending on the project requirements, the daily run data can be reported based on soil "batches" of 152 cubic meters (200 cy) each if desired.

Based on site conditions, changes in selected efficiency values based on changing densities due to changing soil types will be documented. Observations requiring density changes will also be documented in the S3's operational log books.

### 3.4 Energy Calibrations

The Canberra Genie-2000 Gamma Acquisition and Analysis software is used to provide the sorter's standard spectroscopic energy calibration by use of computed energy vs. channel calibration curves. An example of a Genie-2000 derived energy calibration curve is shown in Figure 3-2.

For the detector array design, both the MCNP and ISOCS software suites were used to model and verify the geometry of the NAIS detectors and the soil within the detector's field of view. These models determined the detector array's target energy dependent efficiencies and spatial response profiles. These determinations were made for volumetric sources (i.e. diffuse soil contamination) and for point sources (i.e. discrete radioactive objects). These modeled variables will be compared to the on-site-derived values.

The in-field energy calibration of the S3's detection system for  $^{137}\text{Cs}$  and  $^{226}\text{Ra}$  progeny can be compared to onboard Genie-2000 spectral libraries, and to the photopeaks present in the NIST-traceable  $^{152}\text{Eu}$  calibration verification source.

The cesium photopeak at 662 keV can easily be validated in the calibration spectrum using an exempt quantity  $^{137}\text{Cs}$  check source on the assay belt. As function checking with

one radionuclide is sufficient to validate the energy calibration of the detectors, validation of the other active ROIs with other radionuclides is not required.

Soil depth will be set to match the model depth by the use of the S3's fixed screed system, which also sets the required soil width parameter. Field of view is based on detector height, and the detector array's height above the soils stream will be verified by direct measurement of the belt to detector box vertical dimension. All these set parameters are logged by the S3's historians and written into the data folders for each day's run files. The dimensions will be verified with a ruler prior to commencement of operations and after changes to the geometry, and the evolution will be logged into the S3's operational log.

### **3.5 Traceable Calibration Sources**

Mirion used NIST-traceable sources for all NaI detector response studies. All sources used shall be appropriate for the configuration, type, and target energies of the various radiation emissions relating to the ROCs at HPNS. The S3 calibrations will be documented and certificates for all traceable standards and sources will be provided upon request to the regulatory agencies. The S3 system is calibrated on an annual basis or when the configuration, type, and target energies are changed. Note that the S3 system is response checked daily with traceable and/or non-traceable sources.

Daily function checks on the array's individual detectors are performed with non-NIST 0.25  $\mu\text{Ci}$   $^{137}\text{Cs}$  button sources. Calibration verification and LLRO diversion proofs are performed with NIST-traceable  $^{152}\text{Eu}$  and  $^{226}\text{Ra}$  sources.

### **3.6 ROI Settings**

Different energy ROI are modeled to determine the optimum performance for the detector array. A single broad ROI with an energy range of 10 keV to 1110 keV will be used for gross  $^{226}\text{Ra}$  analysis, as this window is large enough to collect the prominent lines of  $^{226}\text{Ra}$  progeny. This ROI's operational windows are set low enough to capture the uranium  $k$  x-rays and the Compton scattered photons generated by higher energies interacting with the soil and offers additional capability by detecting the higher photon energies from the uranium/radium progeny that are present. Other more focused ROIs will be set to capture individual photopeaks, such as the 609 keV line from  $^{214}\text{Bi}$ . The volumetric efficiency (and MDC) values for  $^{232}\text{Th}$  and  $^{226}\text{Ra}$  in various states of equilibrium (fraction of short-lived daughter progeny relative to  $^{232}\text{Th}/^{226}\text{Ra}$  parent) are presented in Tables 3-4, 3-5, and 3-6.

The S3's detector array will stream all active ROIs to the S3's software platform for logging and analysis as individual or binned detector streams. Volumetric contamination will be monitored using all eight detectors in the array. LLRO detection shall be performed with a separate continuously running algorithm using individual or paired detectors. Both algorithms use identical ROIs (listed below). The ROIs, the widths of the ROIs, and the alarm settings shall not change without Navy and regulator concurrence:

- **ROI 1** shall be initially set at 10 to 1110 keV to collect counts from the majority of the  $^{226}\text{Ra}$  progeny. This ROI will alarm on activity and divert soil to the Diverted Material bin.
- **ROI 2** shall be initially set at 540 to 670 keV to isolate and record the 609 keV peak from  $^{214}\text{Bi}$ . This ROI will alarm on activity and divert soil to the Diverted Material bin.
- **ROI 3** shall be initially set at 625 keV to 698 keV to bracket the 662 keV peak from  $^{137}\text{Cs}$ . This ROI will alarm on activity and divert soil to the Diverted Material bin. Note that compliance with the RG for  $^{137}\text{Cs}$  using the S3 may trigger false-positive alarms due to the presence of other ROCs above normal background levels. If the ROI demonstrates an unsustainable false-positive rate, the Navy may direct ISO to increase the ROI 3 alarm threshold. Compliance with the  $^{137}\text{Cs}$  RG is based on analytical data from systematic soil sampling; therefore an increased alarm threshold does not change  $^{137}\text{Cs}$  screening procedures.
- **ROI 4** shall be initially set at 10 keV to 300 keV to monitor for bremsstrahlung x-ray emissions from  $^{90}\text{Sr}$ . A photopeak occurring in this ROI without an accompanying increase in peak area in ROI 2 will indicate the presence of a strontium source in the soil stream. If this situation occurs, the S3 algorithm monitoring this ROI will alarm and divert this soil to the Diverted Material bin.
- **ROI 5** shall be initially set at 820 keV to 1060 keV to monitor the  $^{228}\text{Ac}$  911 and 969 keV photopeaks when screening material with  $^{232}\text{Th}$  as a ROC. This ROI's alarm will be disabled when  $^{232}\text{Th}$  is not a ROC. When activated, this ROI will alarm on activity and divert soil to the Diverted Material bin.
- **ROI 6** shall be initially set at 2400 keV to 2750 keV to monitor the  $^{208}\text{Tl}$  2615 keV photopeak when screening material with  $^{232}\text{Th}$  as a ROC. This ROI's alarm will be disabled when  $^{232}\text{Th}$  is not a ROC. If used, this ROI will alarm on activity and divert soil to the Diverted Material bin.

### 3.7 Background

S3 array detector (i.e., “instrument”) response to “background” includes contributions from cosmic and terrestrial radiation incident on individual detectors within the heavily shielded S3 Array, as well as the internal electronic anomalies within the detection-counting system itself. “Instrument Background” measurements are typically performed by the S3 with no soils on the assay belt. “Instrument Background” measurements will be repeated after detector-MCA replacement or other major repair, whenever there are modifications to the cosmic/terrestrial system shielding, if the system is relocated to another area of the site, at the discretion of ISO to maintain reliable operations or, at the direction of the Navy.

For the purposes of S3 normal assay operations, net count rates from materials undergoing investigation (after S3 automated subtraction of “instrument background”) are divided by the appropriate efficiency values (Tables 3-4, 3-5, or 3-6) before comparing

calculated activity concentration results against Table 1-1 RG/Background-based diversion criteria. Materials exceeding criteria are diverted for further assessment/management as radiologically impacted.

In addition to typical instrument background, the materials undergoing investigation on the S3 belt will likely contain background ROCs from natural and man-made sources not associated with historic naval site operations. Reference background soil concentration range values for ROCs at HPNS are currently under study by the Navy. When reference background soil concentrations are made available by the Navy, the S3 ROI alarm criteria will be set to the RG criteria (Table 1-1) or the provided background soil concentrations, whichever value is higher (except for volumetric  $^{226}\text{Ra}$  where the alarm will be set to RG) (1.0 pCi/g plus background).

Measurements of instrument background will be determined from an empty belt. The instrument background will be subtracted from the gross project soil gamma results, and net activity in the soil will be determined (pCi/g) for each ROI. Alarms will be set to either the RG or the provided reference background soil concentrations, whichever is higher (except for volumetric  $^{226}\text{Ra}$  where the alarm is the RG plus the background).

Initially, alarm set-points will be based on net activity calculations in the ROIs. For net activity calculations, S3 operating instrument backgrounds will be based on empty main assay belt measurements. Background measurements will be repeated after detector-MCA replacement or other major repair, whenever there are modifications to the cosmic/terrestrial system shielding, if the system is relocated to another region of the site, at the discretion of ISO to maintain reliable operation, or at the direction of the Navy.

### **3.8 S3 Efficiency Determinations**

Prior to assessing minimum detectable concentrations for the S3, Mirion [3] used MCNP modeling to determine typical S3 NaI array detector response in specific ROIs related to  $^{226}\text{Ra}$  (10 - 1,110 keV),  $^{137}\text{Cs}$  (625 - 698 keV) and  $^{232}\text{Th}$  progeny (Ac-228, 820 keV - 1060 keV and Tl-208, 2400 keV to 2750 keV) and to establish instrument efficiency values for diffuse ( $^{226}\text{Ra}$ ,  $^{137}\text{Cs}$ , and  $^{232}\text{Th}$ ) and LLRO point sources ( $^{226}\text{Ra}$ ). Instrument efficiency values were determined for assay belt soil densities ranging from 0.9 to 1.6 g/cc, soil thicknesses ranging from two to three inches (2-3") and are presented as counts per disintegration (c/d) in Table 3-1 (i.e., assuming secular equilibrium and corrected for individual line intensities and applicable branching ratios within the selected ROI). ISO will interpolate ROI efficiency values between density range limits and update the S3 software accordingly, based on ongoing routine assessment of assay belt soil densities and; will apply equilibrium-modified efficiencies (see Section 3.9 Secular Equilibrium) for assessment of diffuse  $^{226}\text{Ra}/^{232}\text{Th}$ . Changes to S3 software efficiency parameters shall remain conservative for the observed soil conditions and be logged whenever modified.

### **3.9 Secular Equilibrium**



Understanding of isotopic parent-daughter equilibrium states is critically important when using gamma interrogation surrogate measurements of photon-emitting daughter products (e.g.,  $^{228}\text{Ac}$ ) to quantify a decay-series parent (e.g.,  $^{232}\text{Th}$ ). Equilibrium corrected instrument efficiency values are presented in Tables 3-4, 3-5 and, 3-6 and, are based on assumptions detailed for HPNS, as follows:

- $^{137\text{m}}\text{Ba}$  ( $T^{1/2} = 2.5$  min) is an intermediary photon-emitting decay product between  $^{137}\text{Cs}$  ( $T^{1/2} = 30.2$  yr) and the ground state of  $^{137}\text{Ba}$  (stable). Given the relationship between parent-daughter half-lives, the 662 keV photon from  $^{137\text{m}}\text{Ba}$  isometric transition is routinely used to quantify  $^{137}\text{Cs}$  under an assumption of secular equilibrium; this is the case for S3 operations where no equilibration corrections are applied to modeled  $^{137}\text{Cs}$  efficiencies in ROI 3.
- Measurements of diffuse  $^{226}\text{Ra}$  (ROI 1) in soil media from surrogate gamma analysis are sensitive to equilibrium states of  $^{222}\text{Rn}$  ( $T^{1/2} = 3.8$  days) as the ROI includes photons the short-lived radon decay products  $^{214}\text{Pb}$  ( $T^{1/2} = 26.8$  min) and  $^{214}\text{Bi}$  ( $T^{1/2} = 19.9$  min). A  $^{214}\text{Pb}:^{226}\text{Ra}$  ratio of 0.5 (Table 3-4) is initially conservatively assumed for instrument efficiency value selection due to the limited potential for radon diffusion within soil interstitial spaces supporting easier emanation and environmental transport away from the original radium source term; for  $^{226}\text{Ra}$  object detection (ROI 2) the physical state lends itself to retention of the daughter progeny and secular equilibrium is assumed for efficiency value selection purposes.
- $^{232}\text{Th}$  is a parcel-specific ROC and may be present above background levels from past military uses in specific areas of the site. The chemical extraction processes to concentrate thorium for commercial/military applications (e.g. production and application of magnesium-thorium alloys) preferentially concentrated thorium isotopes while depleting chemically dissimilar daughter products like  $^{228}\text{Ra}$  ( $T^{1/2} = 5.75$  years) in the final product. Since  $^{228}\text{Ra}$  falls between the ROC  $^{232}\text{Th}$  and subsequent measured daughters including  $^{228}\text{Ac}$ , disequilibrium would affect surrogate gamma measurements based on daughters. However, when the assumption is made that sufficient time has elapsed for secular equilibrium to be restored between  $^{232}\text{Th}$  and  $^{228}\text{Ra}$  (approximately 40 years from initial thorium processing) then, secular equilibrium may be assumed for the remainder of the decay series including  $^{228}\text{Ac}$  (ROI 5), the short-lived thoron ( $^{220}\text{Rn}$ ,  $T^{1/2} = 55.6$  sec) and, subsequent daughters including  $^{208}\text{Tl}$  (ROI 6). Note that branching ratios for isotopes with more than one decay mode ( $^{212}\text{Bi}$ ) are addressed during the modeling of instrument efficiency values for specific ROIs (e.g., correcting  $^{208}\text{Tl}$  photon yields for a 35.9% branching ration).

ISO will obtain Navy concurrence before revising equilibrium assumptions and selecting less conservative equilibration-corrected efficiency values for diffuse scanning.

### 3.10 S3 Activity Calculations

NUREG/CR-5849, Section 8.1.2, Soil Radionuclide Concentration, Equation 8-4 calculates the radionuclide concentration in soil in units of pCi/g. The S3 software calculates the activity concentration on a per second basis, as follows:

#### Equation 2:

$$pCi/g = \frac{27.027 * (gcps - bcps)}{\epsilon * M_{FOV}}$$

Where:

27.027	=	conversion factor (pCi/becquerel)
gcps	=	gross counts per second in the assigned ROI
bcps	=	instrument ROI background in counts per second
$\epsilon$	=	modeled efficiency in units of ROI counts per ROC disintegration
$M_{FOV}$	=	gram mass of soils observed within the field of view in a measurement time equal to one second

### 3.11 S3 Minimum Detectable Concentrations

*A priori* MDC values were calculated by Mirion [3] at the 95% confidence level to ensure sensitivity objectives are met for <sup>137</sup>Cs and <sup>226</sup>Ra (i.e., MDC<RG) using MCNP-modeled efficiencies and the following adaptation of the “Curie” MDC formula:

#### Equation 3:

$$MDC \left( \frac{pCi}{g} \right) = \frac{27.027 * (k^2 + 2 * k * \sqrt{2 * N_B})}{\epsilon * T_B * M_{FOV}}$$

Where:

27.027	=	conversion factor (pCi/becquerel)
$k$	=	coverage factor (1.645 for 95% confidence)
$N_B$	=	instrument ROI background total counts
$\epsilon$	=	modeled efficiency in units of ROI counts per ROC disintegration
$M_{FOV}$	=	gram mass of soils observed within the field of view in measurement time
$T_B$	=	( $T$ ) background measurement time in seconds

Note: To simply calculate minimum detectable activity (in  $\mu\text{Ci}$ ) using Equation 3, replace the soil mass within the field-of-view factor with the conversion factor ( $1\text{E}6 \text{ pCi}/\mu\text{Ci}$ ).

Minimum detectable concentrations for diffuse  $^{226}\text{Ra}$  and  $^{137}\text{Cs}$  are presented in Table 3-2. For  $^{226}\text{Ra}$ , Table 3-2 represents secular equilibrium and Table 3-4 represents other equilibrium states. Minimum Detectable Activities for LLRO  $^{226}\text{Ra}$  are presented in Table 3-3. Calculated MDC/MDA values are below target values for belt speeds up to seven inches per second, for belt soil thicknesses between two and three inches, and for soil densities ranging from 0.9 to 1.6 g/cc.

### 3.12 S3 Alarms

The S3 software generates a second-by-second database of the calculated activity results for each detector in every ROI. The data array for a single ROI takes the form of a series of columns, with the first column, Column A, automatically registering the date and time every second. Column B (the second column in the database, as the S3 uses zero indexing) presents the pCi/g activity output from Detector 1, while Column C presenting the activity output for Detector 2, etc. Note that every row in the array represents one second in time, with Row 1 being the most recent value.

After the columns of calculated data, another column outputs the maximum value from all detectors in the array. The S3 software monitors and compares that latest value (in pCi/g) to the stored Distributed Alarm value for that ROI.

As an example, in ROI 1, the alarm value is entered and saved by the S3 as the value that equals 1 pCi/g plus background. If the maximum value (in net pCi/g) in the database's Row 1 equals or exceeds this alarm level, the S3 flags the row and initiates a diversion sequence while continuing to monitor the length of time that that and any other column's value is equal to or above the alarm level.

With the exception of  $^{226}\text{Ra}$ , the Navy will use the RG or reference background soil concentration, whichever value is higher, as the project action limit. The project action limit for  $^{226}\text{Ra}$  is the RG plus background.

Other columns in the data array collect related operational data, including the S3's current diversion status, the current direction of travel of the S3's reversing diversion conveyor, the pause state of the assay belt, and columns of proprietary information.

### 3.13 Alarm Summary

To reiterate, the RGs for each ROC at HPNS is captured by the sorting system as alarm/diversion set-points;

- ROI 1, which is the broad  $^{226}\text{Ra}$  ROI, will be set to 1.0 pCi/g above background.

- ROI 2, monitoring the  $^{214}\text{Bi}$  609 keV peak, will be set to 1.0 pCi/g.
- ROI 3, monitoring the  $^{137}\text{Cs}$  662 keV peak, will be set to 0.113 pCi/g.
- ROI 4, if used for detection of  $^{90}\text{Sr}$  LLROs, will divert all activity distinguished by the system using the special detection algorithm (see Section 5.2).
- ROI 5, when monitoring the 911 and 969 keV photopeaks for  $^{228}\text{Ac}$ , will be set to 1.69 pCi/g.
- ROI 6, if used, will monitor the 2615 keV photopeak for  $^{208}\text{Tl}$ , and will be set to 1.69 pCi/g.
- For  $^{226}\text{Ra}$  LLROs, the alarm setpoint is 0.087  $\mu\text{Ci}$ .

When background soil concentration values are made available by the Navy, the S3 ROI alarm criteria will be set to the RG criteria (Table 1-1) or the provided background, whichever value is higher (except for  $^{226}\text{Ra}$  where the alarm will be set to the RG plus background).

## 4.0 PRE-OPERATIONAL TESTING AND OPERATIONAL CHECKS

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### 4.1 <sup>226</sup>Ra Object Detection Verification

Detector efficiencies were modeled in MCNP for a point source of <sup>226</sup>Ra (in secular equilibrium with daughters) on the S3 conveyor belt covered by otherwise uncontaminated soils in an off-set position between two detectors. LLRO <sup>226</sup>Ra MDAs were then calculated across a range of soil densities and belt soil thickness based on the modeled efficiencies under worst-case expected source-to-detector geometry conditions (See Section 3.11). Note that previously screened site or approved clean import fill soils may be used as LLRO carrier material and for instrument backgrounds during the object verification.

After mobilization, LLRO detection verification will be performed by ISO using a nominal 0.087  $\mu\text{Ci}$  <sup>226</sup>Ra point source at the established LLRO <sup>226</sup>Ra ROI 1 diversion alarm setpoint. The performance verification steps are as follows:

1. The source container is first placed directly on the belt between two detectors and covered by otherwise uncontaminated soils to emulate the worst-case modeled source attenuation characteristics.
2. The CRO will process the soils using the planned operating parameters; a positive diversion alarm in ROI 1 is considered a successful test.
3. The process will be repeated 29 additional times (30 total), repositioning the source in a reasonably distributed fashion at various depths and points along the 4.75-foot wide soil stream width for each subsequent pass, including the extreme edges of the soil stream.
4. The outcomes of the 30 test passes are evaluated as follows:
  - a. If the success rate after 30 tests is  $\geq 95\%$  (i.e, no more than a single miss), the S3 has met the <sup>226</sup>Ra LLRO sensitivity measurement quality objective.
  - b. If the success rate is initially  $< 95\%$ , up to 70 additional test passes will be performed, at the discretion of ISO, to demonstrate a success rate  $\geq 95\%$
  - c. If after 100 tests (or earlier at the discretion of ISO) the success rate remains  $< 95\%$ , ISO, in collaboration with the Navy and APTIM,, will use professional judgment and reduce the Soil Belt Speed/Depth parameters and repeat the entire process (potentially multiple times) until detection and diversion are demonstrated at the 95% confidence level with a minimum of 30 test passes at the final S3 operating parameters.

## 4.2 Distributed Contamination Calibration Verification

ROI alarm set-points for ROCs are based on the consideration that the true contamination may be discrete or distributed. Modeled efficiencies were determined under both scenarios.

1. For volumetric calibration verification, ISO will model typical S3 detector efficiencies using a volumetric NIST-traceable broad-energy  $^{152}\text{Eu}$  gamma source. This specific source will be used for subsequent volumetric field test evaluations. The  $^{152}\text{Eu}$  source density will be developed with a conservative target density of ~1.6 g/cc for consistency with the maximum expected soil density and a nominal activity of 5  $\mu\text{Ci}$ .
2. The modeled detector efficiency for the Eu source within the  $^{226}\text{Ra}$  ROI will be compared to measurement(s) performed after S3 setup with the same source and source-detector geometry, as modeled (e.g., stationary on the belt with no other soils present).
  - a. If the observed detector efficiency for a representative detector falls within 30% of the modeled efficiency for a typical detector (inclusive of all possible sources of bias and uncertainty in the efficiency calculations), the ability of the S3 to meet performance objectives using modeled detector efficiencies for volumetric sources will have been positively demonstrated. The “+/- 30%” range is selected based on “Calibration Confirmation Acceptance Limits for NDA Systems” referenced in the Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Revision 5.3, May 2019 (Appendix D, “Non-Destructive Assay”, Table D-1)[12]
  - b. Should the objectives not be met, reviews of energy calibrations, gain matches, and other calibration parameters shall be investigated first, prior to hardware removal and replacement.

The S3's belt speed and soil thickness will be initially set based on the values that yield calculated *a priori* distributed MDC values below the RGs for gamma-emitting ROCs. ISO, in consultation with APTIM and the Navy, reserves the right to modify scanning conditions, as needed, provided that modeled efficiency values are available and *a priori* MDCs recalculated for gamma-emitting ROCs remain below the applicable diversion criteria. At the conclusion of the pre-operational testing, the MDCs will be recalculated using actual site- and instrument-specific data.

## 4.3 Daily S3 Function and Source Response Checks

Source response checks for the S3 are measurements performed to verify detector performance against a known standard daily during normal operations; the detector array is function-checked during startup for each day's use with the 'S3 Source Board', an array

of identical non-traceable 0.25  $\mu\text{Ci}$   $^{137}\text{Cs}$  sources mounted on a flat plate holder that is placed in a reproduceable geometry under the detector box, which produces a response in each array detector. The daily source response check acceptance range is  $\pm 30\%$  from average values derived after pre-operational testing and before commencement of sorting operations. In addition, the S3 operators may perform additional qualitative response checks throughout the day to verify the system detection and diversion processes are functioning properly and are documented in the S3's daily log when performed.

Daily quality assurance/quality control records for each detector combination are automatically documented by the S3 along with the calibration certificates and instrument setup test records. This documentation is captured by one of the historians built into the S3's software. The historians record the daily control chart data and many other aspects of the system; history output options include ASCII text or Excel-readable comma-separated value files that are automatically recorded into their own directory on the system's computer hard drives.

If an NAIS detector, Osprey MCA, or a related support component is found to be in a questionable physical condition, that component will not be used until the condition has been corrected. If the condition cannot be corrected, or the component has exceeded its annual calibration date without approval, it shall be placed into an 'out-of-service' state. This is done by placing an *out-of-service* tag or equivalent on the component and securing it in a separate area such that it cannot be used until the out-of-service condition has been rectified.

If a NAIS/MCA detector combination fails an operational check, the prescribed corrective action involves repeating the measurement. If the repeated measurement is again out of its control limits, the S3 CRO will stop operations, remove and tag the instrument out-of-service, and notify the ISO-Technical Lead for corrective actions based on professional judgment up to replacement of the detector/MCA set(s) in question. The steps described in Sections 3.6 and 3.9 are then repeated for the new or repaired detector/MCA combination prior to return-to-service. In addition, background and new daily source board measurements are then conducted to verify MDCs against applicable RG and to support future daily function and source response checks. Documentation of the detector/MCA servicing event and the corrective evolution are captured in the S3's Daily Log by the CRO and reviewed and approved by the ISO-Technical Lead.

#### **4.4 S3 Control Charts**

The S3's control charts document the performance of the measurement system during and over a period of interest. The control chart is a graphical plot of measurement results with respect to time or sequence of measurement, together with limits within which the measurement values are expected to lie when the system is in a state of statistical control. Calibration and QA check results are plotted on control charts, and these charts are developed for response measurements where the expected performance is established, monitored, and documented.

*Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM)* states that a separate set of control charts for monitoring each type of measurement should be developed for each instrument. ISO has developed control charts for S3 functional one-minute long source board checks and for ten-minute long empty belt background counts for all eight NAIS detectors. These apply to pre-operational calibration periods and post-calibration operational periods.

The S3's control charts are constructed as a graph showing the arithmetic mean and the control limits as horizontal lines. The control limits upper and lower boundaries marked as plus/minus 30% of the mean. The measurement results in the appropriate units are shown on the y-axis and time and sequence are plotted using the x-axis. Other control limits can be used if needed. An example of an S3 control chart is shown in Figure 3-3.

As the background counts and source board counts are performed, the results are entered on the control chart automatically by the software. If the results are outside the control limits or show a significant trend, then the process is not in control and the ISO-Technical Lead is notified by an on-screen message. Provided the routine performance is within the control limits, the results obtained using that instrument are acceptable and can be used for assay and sorting.

The "CHARTS" page of the S3 software is the gateway to the control charts for the detection system. There are four charts created for every detector:

1. Channel x background control chart – Initial Calibration
2. Channel x background control chart – Operational
3. Channel x source board control chart – Initial Calibration
4. Channel x source board control chart – Operational

Where:

x = the detector number, using the counts from the Prime ROI

Operationally, the CRO can elect to save the last one- or ten-minute's worth of data points for an individual detector, or, with approval from the ISO-Technical Lead, to delete the entire history for that detector. This function is only used when a detector has been changed out and new control charts need to be run for the new detector. This evolution is documented in the S3 Logbook and reported in writing to the PRSO.

When control charting for background, outside factors can cause the S3's Scan MDC's to exceed the RG. Examples include 'shine' from nearby feed piles, nearby above-criteria piles, etc. Unless the calculated activity resulting from outside background disturbances like these exceeds the Scan MDC, no corrective actions will be taken regarding background trending and tracking in these scenarios: however, the PRSO will be notified of the suspected conditions.



Trend analysis of the control charts for the  $^{137}\text{Cs}$  source board array will include the following observations and corrective actions, among others as directed in the S3 Operating Procedures:

- a. **One point out of the control chart limits of the mean plus or minus 30%.** Corrective action involves repeating the measurement. If the repeated measurement is out of control limits, stop and tag the instrument out-of-service and notify the ISO-Technical Lead. If the repeat test is within control chart limits, continue using the instrument.
- b. **Eight consecutive points are on the same side of the mean.** Corrective actions include ensuring that the source board is correctly positioned for the source count and that there is no obvious physical damage to the board, the source, or the detector instrument. Provide the information to the ISO-Technical Lead for evaluation and perform additional test measurements as directed.
- c. **Eight consecutive points are arranged so each point is larger (or smaller) than its immediate predecessor.** Corrective actions include ensuring that the source board is correctly positioned for the source count and that there is no obvious physical damage to the board, the source, or the detector instrument. Provide the information to the ISO-Technical Lead for evaluation and perform additional test measurements as directed.
- d. **An obvious cyclic pattern seen in the data points.** Corrective actions include providing the information to the ISO-Technical Lead for evaluation and perform additional test measurements as directed.

Note that the ISO-Technical Lead may alternatively direct the use of other database programs for charting purposes in lieu of the S3's onboard charting functions. This is particularly true when using the complex Multi-ROI operations.

## 5.0 LLRO RETRIEVAL AND SAMPLING EVOLUTIONS

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### 5.1 Object Retrieval from the Assay Belt

When the S3's sampling function is activated, the assay belt will automatically stop a source (typically an LLRO) at the assay belt's sampling point for manual retrieval by a supporting RCT trained to use appropriate handheld survey meters and safely handle radioactive materials according to the radiation protection plan. Once stopped, the system will not resume processing until a command is received from the CRO.

The S3's sampling point is downstream of the detector box and is marked in yellow on the conveyor framework to facilitate locating the suspect materials. The sampling point is shown in Figure 4.1. While the S3 will automatically report which detector identified the LLRO, and this information will be shared with the sampling technician, he/she may also utilize a handheld radiation detector to supplement their effort of locating and retrieving the suspect material. It is possible for a person of average stature to reach the middle of the S3's assay belt from the catwalks on either side of the conveyor structure.

The S3 will generate a waste information log entry for each event to facilitate archival recording and disposal or transfer of discovered items. A numerically organized and timestamped log entry details the radionuclide identification (via ROI) and estimated curie content of each LLRO. The log files are created immediately following observation of the suspect material, and no input or intervention is required from the S3 operators. At the end of each processing day, ISO provides the client with an electronic file of each batch's run data, and a file folder of all suspect material discovered in each batch are part of the sorting process documentation.

Note that other modes of sampling can be performed at the assay belt's sampling point, including collection of biased, unbiased, and systematic soil samples as directed by the final project-specific work plan for the parcel. Coordination with the system operators is required to stop the S3 at a desirable time, and if sample number logging is desired, that information can be relayed to sampler to be recorded, as it is on the electronic date- and timestamped S3 Operations Log.

### 5.2 Strontium Marker Detection

High-energy  $^{90}\text{Sr}$  beta particles interact with high-Z material such as soil, rock, steel, and possibly even the S3's titanium and lead shielding materials causing minor variations in angular momentum. The resulting "bremsstrahlung" or "braking radiation" yields a spectrum of photons with energies ranging from near-zero up to the maximum kinetic energy of the incident particles (546 keV), as shown on the S3 spectral screen capture in Figure 4-2. The counts from the NAIS detectors in ROI 4 may serve as a method for indirect detection of  $^{90}\text{Sr}$  radioluminescent markers; the potential for false positives in this specific ROI is higher given that the presence of  $^{226}\text{Ra}$  objects will create Compton photon scattering effects that will increase the count rate in ROI 4

Considering the average beta energy (195.8 keV) and expected distribution, approximately 90% of the available photons will occur within the 100-300 keV ROI. By limiting the upper window ROI threshold to 300 keV, the potential impact from <sup>226</sup>Ra daughters such as <sup>214</sup>Pb (351 keV) is minimized with an additional benefit of reduced background without significant impact to instrument efficiency.

### **5.3 Timed Soil Sampling Activities**

If project-specific work plans call for systematic samples to be collected from the S3's sampling point at a given period, the frequency of sampling is determined to provide a systematic distribution of sample collection throughout each survey unit. The required number of systematic samples collected per batch will be determined in the project-specific work plans and SAPs. The project-specific work plans describe the evolution using an example, where if the S3 is configured to process a 200 cubic yard (152 cubic meter) batch and 18 systematic samples are required, a systematic sample will be collected for every 11 cubic yards processed.

Samples will be collected from the assay belt with hand tools in accordance with the project SAP. The S3 software is configured to present a visual reminder and a sound reminder for a sampling interval that is due based on the actual production time or volume summary. This feature of the software alleviates the need to manually track production or operation times.

All non-ISO personnel involved with these operations shall coordinate their sampling evolutions with the S3 operators.

### **5.4 Transfer of Excavated Soil to the S3 for Processing**

A volume target batch of approximately 200 cubic yards (152 cubic meters or survey unit equivalent) of soil will be delivered to the S3's Staging Area with a Truck Ticket with the following information:

- Location of excavation or former trench name from which excavation, excavation soil unit (ESU), or SFU material was excavated (as applicable)
- Load number
- Estimated volume of soil
- Date and time of excavation

The S3 CRO will record this information, and the S3 will automatically assign a filename for the batch. This information will be logged automatically by the S3 via a historian function, and the filename and the batch information will be inseparably linked by the S3 software.

Automatic batch file logging by the S3 will include the following typical naming formats:

- EX##-S3-XXX for Parcel E excavation material (with remedial excavation identification number and sequential sample number identified)
- HPPG\_ESU\_XXXX for Parcel G excavation and/or former trench backfill material
- HPPG\_SFU\_XXXX for Parcel G sidewall and bottom material from trenches

Note that 'ESU' references an excavation and/or former trench backfill soil unit, while 'SFU' references a trench sidewall or floor unit.

Following completion of a survey unit, ESU or SFU batch, the S3's radiological results will be generated using the S3's software. Reports will include the basic statistical metrics for each of the two 'bins' of soil that were created including second-by-second logging of raw count rates and calculated activity for each detector, and all associated static variables, such as reference background, belt speed, soil layer thickness, soil density, efficiency information, total number of data points in the data set, and others. These constants are captured in a S3 parameter record historian and the variables are reported with the daily run data. There can be any number of individual data historians in the S3 software suite.

All soil processed by the soil sorter system will be staged pending evaluation of off-site analytical results and Navy direction regarding final disposition (off-site disposal or on-site re-use). A layout of the S3 in typical configuration is shown in Figure 4-3.

## **5.5 Above-Criteria Sampling Evolutions**

When above-criteria soil material is discharged to the Diverted Material bin, an investigation of the potential area of elevated activity in the bin will be conducted by site RCTs. At a minimum, the results from the soil sorter's reporting software will be reviewed to identify the causes for diverting material, and biased soil samples will be collected. Biased soil samples may be collected from the belt prior to diversion or from the soil discharged to the Diverted Material bin at a minimum frequency equal to the volumetric frequency of sampling for a survey unit, ESU or SFU batch, in accordance with the project-specific work plan sampling procedures.

## 6.0 DATA COLLECTION AND REPORTING

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### 6.1 S3 Data Collection

All S3-generated electronically recorded data and information will be backed up to a SharePoint site or local equivalent on a nightly basis, or as reasonably practical.

The list of data folders created by the S3 historians are as follows:

- **Batch Report** – This folder contains a summary of the batch filename, including
  1. Production Details, listing:
    - a. Above-Criteria Volumes in both cubic yards and tons,
    - b. Below Criteria Volumes in both cubic yards and tons,
    - c. Total Material Volumes in both cubic yards and tons,
  2. Samples Taken, listing:
    - a. Sample Number, Sampler's Name, and a timestamp in hh:mm:ss
    - b. Sample Interval in cubic yards
  3. Below Criteria Concentration in pCi/g, listing:
    - a. Mean +/- UCL95
    - b. Min
    - c. Max
    - d. Number of observations
  4. Above-Criteria Concentration in pCi/g, listing the same information above:
- **Data Charts** - This folder contains a series of charts from the latest batch of soil processed. The charts contain color-coded pCi/g values from each detector, plotted with activity in the y-axis and process time in the x-axis. Superimposed over the data is a red horizontal line that represents the RG level for that ROI. An example is presented in Figure 5-1.
- **Control Charts** – this folder contains the daily updated set of control charts for every detector. The control chart folder includes charts for each detector's empty belt background and functional response counts with the S3 Source Board. Note that the upper threshold for background charts will be what the ROI background count rate would have to be to exceed the RG for that ROI. Any exceedances call for corrective action, such as detector status investigation, system cleaning, determination of potential nearby sources, etc.
- **Dist\_Activity** – this folder contains the result of the distributed soil activity calculations performed on the raw data, for each active ROI.
- **Efficiencies** – this folder contains a list of each detector's current efficiency for each ROI.

- **Error\_Log** – This folder contains a record of all faults generated by the S3's code monitors and is used by ISO to facilitate troubleshooting.
- **Log\_Book** – This folder contains a record of all entries made by the software and by operating personnel. In addition to the multiple timestamped records generated by automatic processes and other actions taken by the S3 software, the CRO will supplement the record by electronically logging all information about events that impact operation of the system. Every action taken by the system or by the operators is automatically timestamped and logged. The names of all personnel operating on or stationed with the S3 are recorded electronically in the S3 data files.
- **Object\_Log** – this folder contains the Object Log. This is where data regarding the data capture of high-activity potential LLRO's is logged. The log consists of a time stamp, object number, alarming detector number, alarming ROI, and activity level in curies.
- **Parameters** – This folder contains a record of all current variables and constants used in the sorting process. They include:
  - a. the object sorting criteria (for HPNS, the 226Ra object RG)
  - b. the distributed soil sorting criteria (the volumetric RGs for each ROI)
  - c. the soil depth setting
  - d. the soil width setting
  - e. the soil density setting
  - f. the belt speed setting
  - g. the mass of soil in a single detector's field of view
  - h. the detector efficiencies for distributed contamination by ROI
  - i. the detector efficiencies for LLRO contamination by ROI
- **Raw\_Data** – this log records the raw output of all detectors, in cps, for every ROI, from software startup to shut down. All data is time stamped by the software's high-precision clock.
- **Reference\_Bkg** – this folder contains a log of the fixed reference background values for each detector and ROI in counts per second.
- **Sources** – this folder contains a table of all sources used in the calibration of the S3. The table contains the source ID number, the nuclide, birthdate, birth activity, half-life, manufacturer, and the current decayed activity in  $\mu\text{Ci}$  or  $\text{pCi/g}$ .
- **Statistics** – this folder contains a log of the more advanced statistical metrics for each of the two bins of soil that are created. This log is produced for each ROI.
- **Temperatures** – this folder contains a log of the current temperature inside the detector box and the external temperature, captured every minute.

- **Thresholds** – this folder contains a record of the current alarm levels (RGs) for every ROI.
- **Volumes** – This folder contains a summary of the soil volumes processed by the latest sorting campaign. Each bin (Diverted Material and below criteria) is summarized in cubic yards.

All records of field-generated electronic data developed by the S3 will be reviewed first by the ISO-Technical Lead or his designee, and then by the PRSO or a designee knowledgeable in the measurement method for completeness, consistency, and accuracy. This extensive soil screening data package is developed for each 200 cubic yard batch processed and contains all gamma scan measurements, figures, logged parameter data, a suite of statistics, and all summary tables.

On-site electronic copies of original electronic data sets will be preserved on a nonmagnetic retrievable data storage device, such as USB drives or an optical drive. Data and information recorded on paper will be recorded using indelible ink. No data reduction, filtering, or modification will be performed on the originally submitted electronic versions of S3 data sets.

## 6.2 Human – Machine Interface

The S3's Waterfall page is perhaps the most important graphical aid available to the CRO. Displayed here are two waterfall plots, one 30 seconds long and angled in perspective so that it visually represents the sorter belt, and another, 10 minutes in length, which can highlight soil activity patterns, detector response variations, and external radiation influences over time. This page is shown in Figure 5-2.

The plot on the left-hand side of the screen, *NET GAMMA ACTIVITY DOWNSTREAM OF DETECTOR ARRAY*, is the primary tool for visualizing radioactivity in the soil stream. The CRO can also visualize the random background fluctuations in the data stream while the sorter belt is stopped.

The waterfall plot data is arranged so that it appears to be moving at the same rate as the belt speed. The top of the waterfall graph is where the detector box resides, and the bottom of the graph represents the point of departure on the assay belt, the place where soil begins to fall on to the diverter belt. Each column of 'tiles' corresponds with the number of detectors in the array, and each 'tile' represents one second of output data for that detector. Note that in the array shown there are eleven columns of data. In the S3 constructed for work at HPNS, there are eight detectors, thus there will only be eight columns of data.

The right-hand waterfall, *NET GAMMA ACTIVITY ALL CHANNELS TEN-MINUTE ARRAY*, is used to provide a more historical visual record of activity. The plot is sensitive enough to alert the CRO to radiation shine from nearby soil piles and other sources. It

can also alert the CRO to a repeating pattern, whether from a low or high counting detector, a poorly graded division of soil particle size on the sorter belt (for example, gravel on one side, fines on the other) or a repeating signal from a discrete source stuck on the sorter belt, or an inaccurate detector efficiency.

A normal presentation of this plot could be compared to television static. When looking at an empty belt background, for example, if no pattern exists, that is, if the plot field appears to be random noise, then this is a favorable state. Higher counts, trending towards a red color on one side of the plot or the other, may indicate shine from a nearby source, and the CRO will investigate.

These waterfall plots are automatically set to display the Prime ROI. No data can be seen from other ROIs unless the CRO switches to the desired ROI. The schema used for both plots is designed to highlight activity using red against blue and dark gray backgrounds. The values assigned to each color may be edited by using the Schema Editor.

When desired, the S3 will identify the next incoming higher-activity object. This function identifies the next object that is above the RG criteria. To capture the next particle, the CRO will click the *CAPTURE NEXT* button on the waterfall page.

### **6.3 Documentation Quality Standards**

All S3 data channels can be displayed and graphed but can never be modified or manipulated as it is physically impossible for anyone to change or to manipulate the saved values on any channel of data. Program editing security is controlled by a high-level management password which is required to open the program document in any version of our software. A physical USB crypto key must also be connected and recognized by the S3 computer for the S3 program to operate.

The S3 has several built-in safeguards to make certain the process is under control. The system's code is written so that any abnormal communication event, parameter change, or attempt to edit a value triggers an automatic 'All Stop' sequence that shuts down the system and generates an error report. Other coded safeguards include timestamp comparisons that cross-check current data and pre-programmed variables, scans for empty arrays or not-a-number values, and others. To foster transparency in S3 operations, the S3's Error\_Log subfolder contains these entries.

The S3's process computer is a multiple-core industrial fanless computer, with dual redundant power supplies. Equipped with onboard uninterruptable power supplies and second-by-second data recording, process data cannot be lost due to power failure.

To augment this capability, the software also uses an automatic file splitting protocol. The Auto Split Files function determines the number of rows written per file and is useful for long-term data acquisition to avoid creating excessively large files and to prevent data loss in case of file corruption. This protective feature of the software creates multiple sequential data files for each channel being logged, which are closed at a preset file size



interval and a new file automatically opened. Auto Split properly organizes the files by appending the date and time of each file creation to the master file name.

All process data is exported to its respective log on the hard drive every second and stored for later download. To ensure traceability, each day's master log record will begin with an entry of the following information:

- Name of the project
- Specific location
- Function and process
- Date
- Document number (if applicable)

The quantities used in S3 records are clearly indicated in standard units (e.g., curie, cps, cpm, dpm, becquerel), including multiples and subdivisions of these units.

#### **6.4 Record Retention**

Records resulting from implementation of this work plan will be retained as outlined in the project-specific work plans and associated appendices.

## 7.0 REFERENCES

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8. ISO-01 Rev 1.6, ISO-Pacific S3 Operations Manual for Version 1.2.6
9. Genie-2000, S502 Basic Spectroscopy Software for Single Input Applications
10. Canberra Osprey Universal Digital MCA Tube Base 7066438B User's Manual
11. ASTM D75 / D75M-19, Standard Practice for Sampling Aggregates, ASTM International, West Conshohocken, PA, 2019
12. Department of Defense/Department of Energy Consolidated Quality Systems Manual for Environmental Laboratories, Revision 5.3, May 2019 (Appendix D, "Non-Destructive Assay", Table D-1)
13. ASTM D7204-15, Standard Practice for Sampling Waste Streams on Conveyors, ASTM International, West Conshohocken, PA, 2015
14. CB&I Federal Services LLC, 2015, Final Technical Basis Document, Gamma Scanning Surveys, Former Naval Station Treasure Island, San Francisco, California.

## Figures

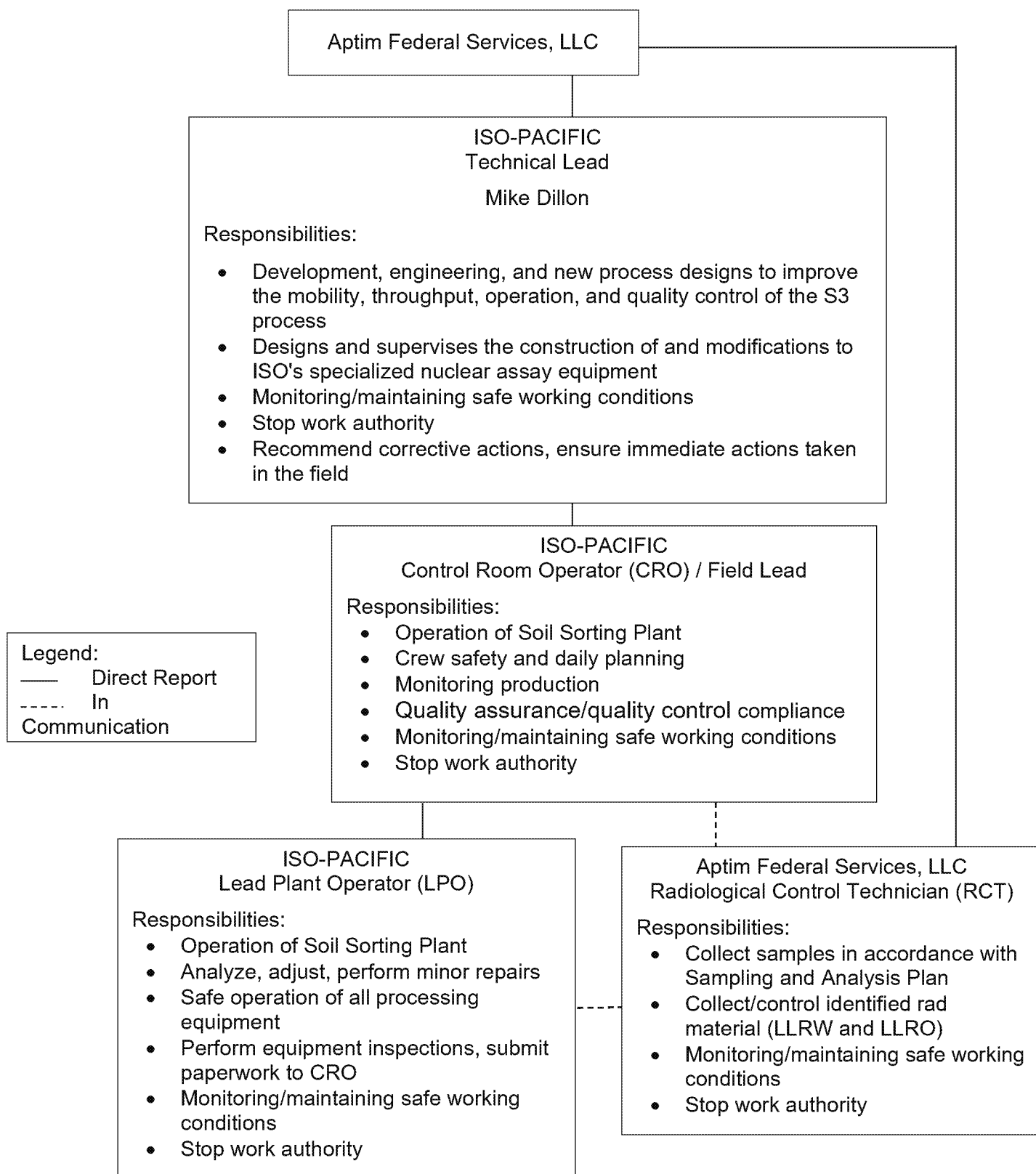


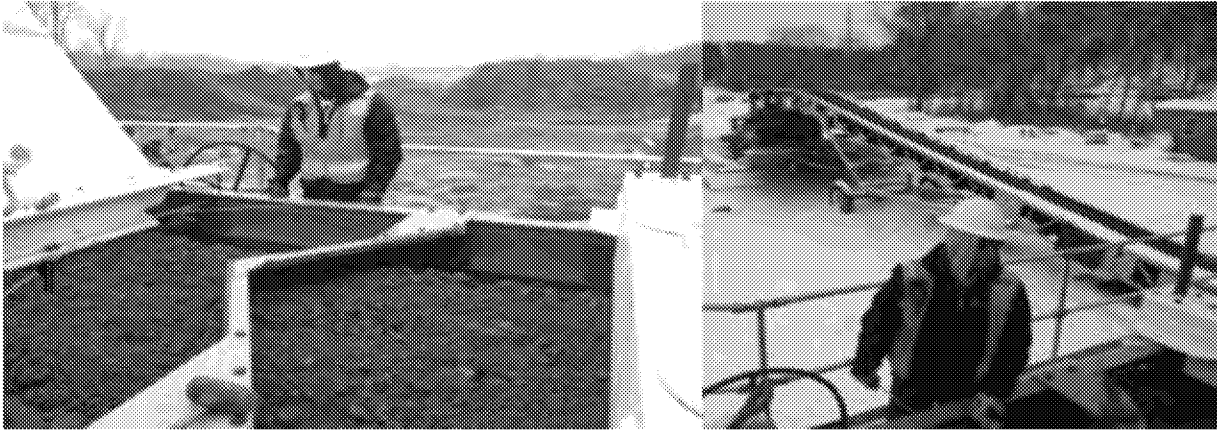
Figure 1-1. Organization Chart and Role and Responsibilities



Figure 1-2. S3 operations in the rain



**Figure 1-3. View from the S3 Control Room**



**Figure 1-4. Inclement weather and extreme cold conditions**

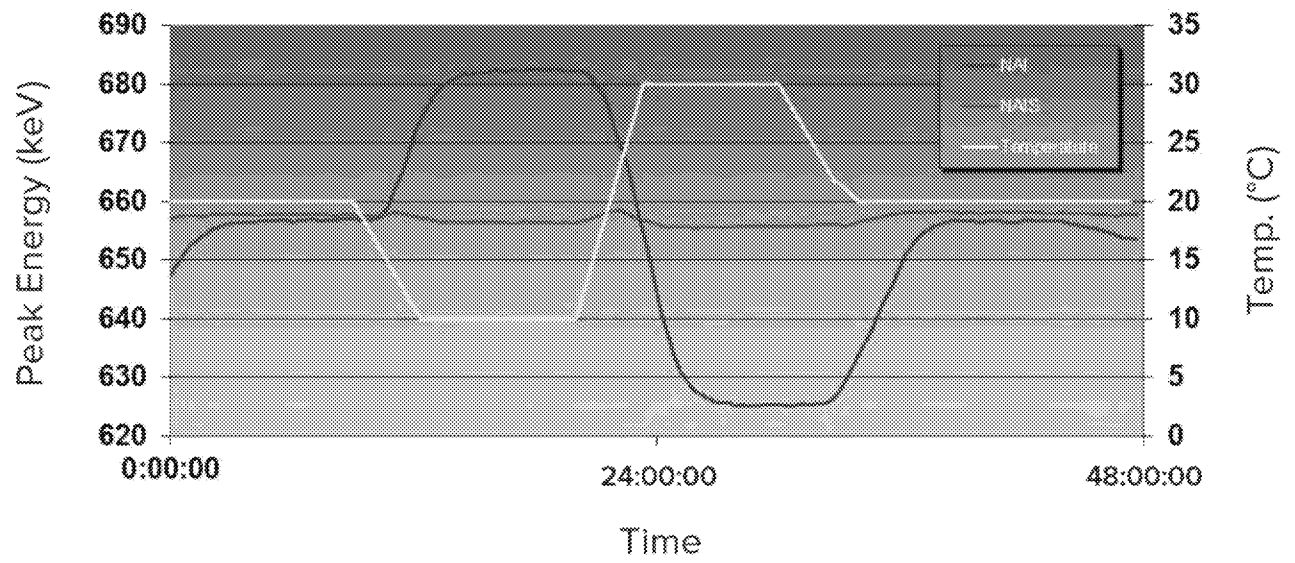
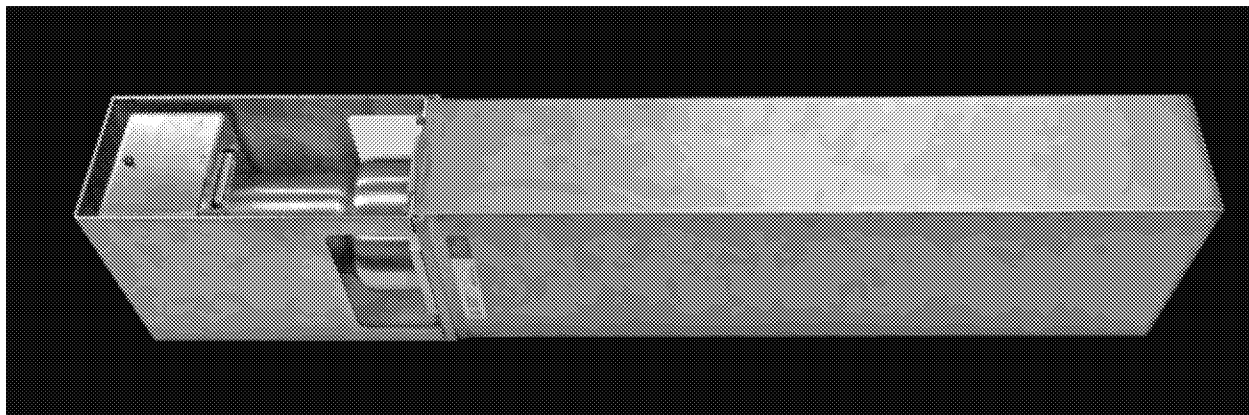


Figure 2-1. NAIS centroid variation for temperature range 10 -30 °C





**Figure 2-2. Mirion Canberra Model NAIS 3x5x16 Temperature Stabilized Sodium Iodide Scintillation Detector**

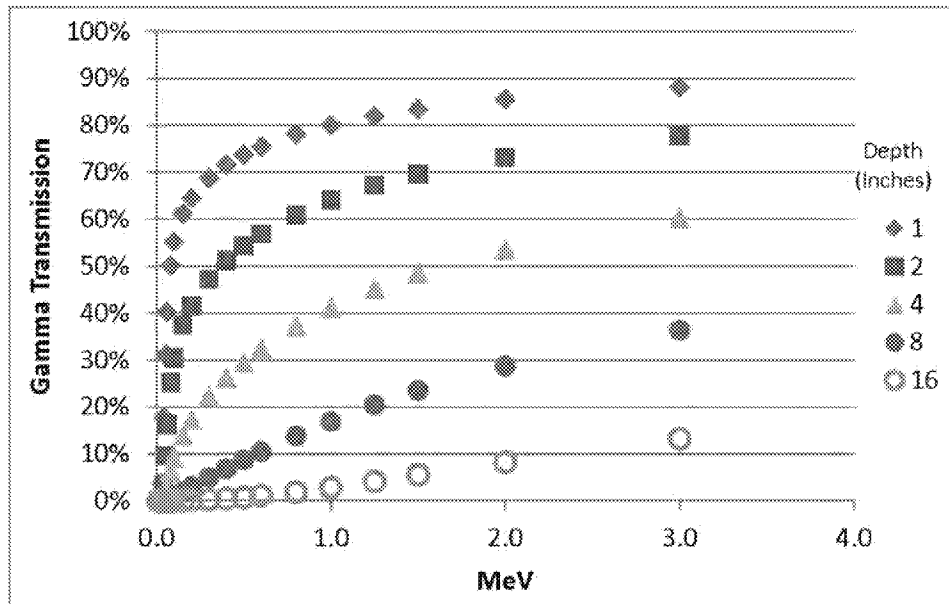


Figure 3-1. Gamma Transmission as a Function of Energy and Soil Depth  
(HydroGeologic, 2012)

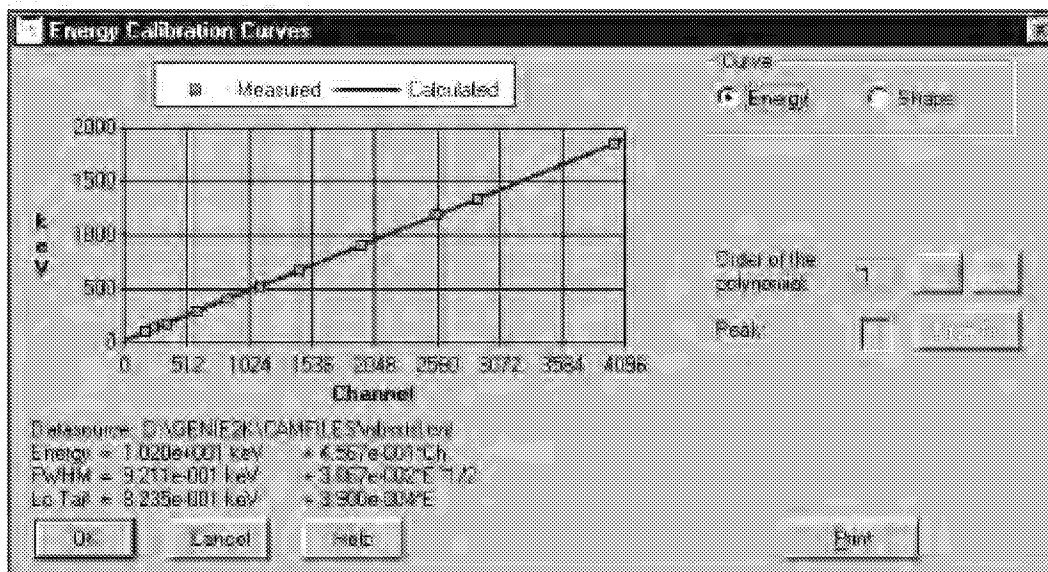


Figure 3-2. An S3 Energy Calibration Curve developed by a Genie-2000 library file

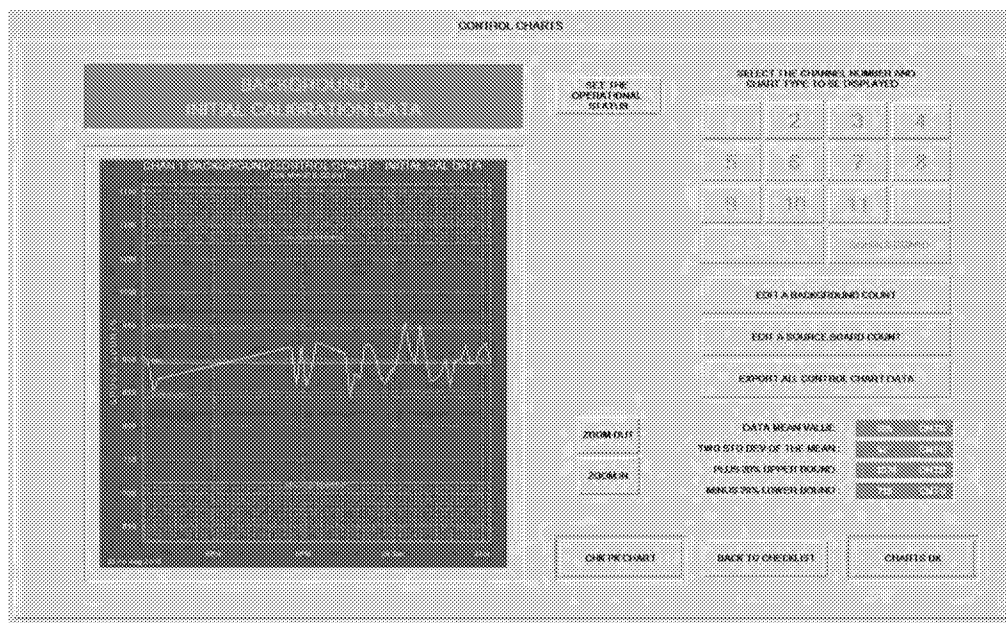
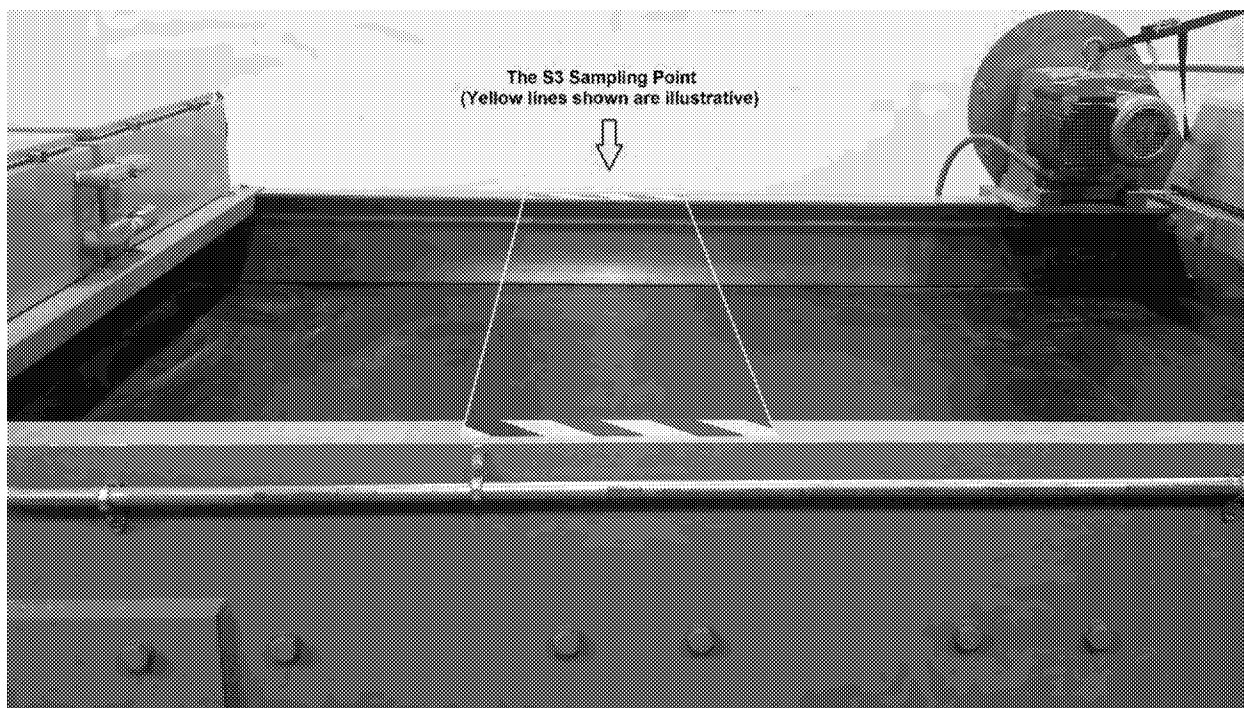


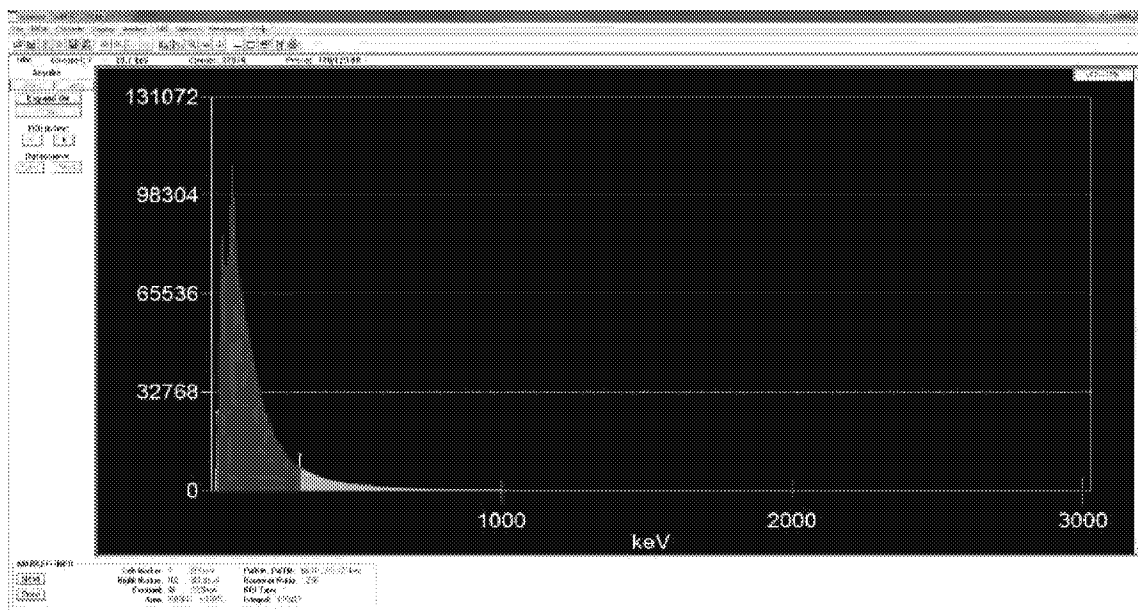
Figure 3-3. Control Chart for S3 Detector 1, Initial Calibration Data for Empty Belt Background



**Figure 3-4. An example of a belt sampling template used in on-conveyor soil density determinations  
(Photo Is Fig. 1 From The ASTM D75/D75M Document)**



**Figure 4-1. The S3's Sampling Point on the Assay Belt**



**Figure 4-2. The S3's bremsstrahlung spectrum resulting from 90Sr beta Emission In Soil And Interaction With The System's Soil And High-Z Materials**

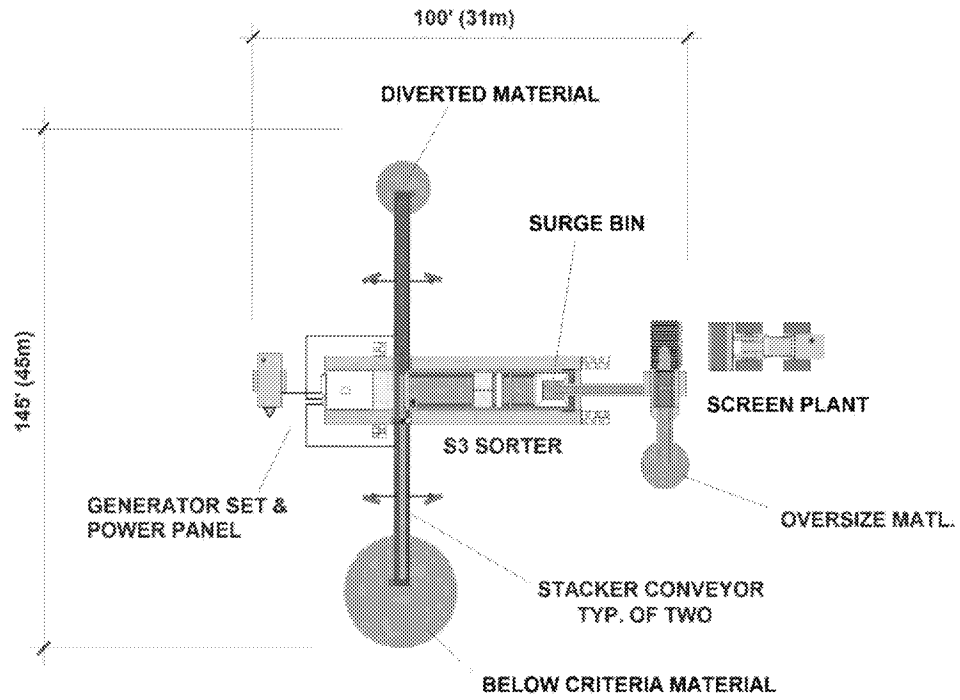


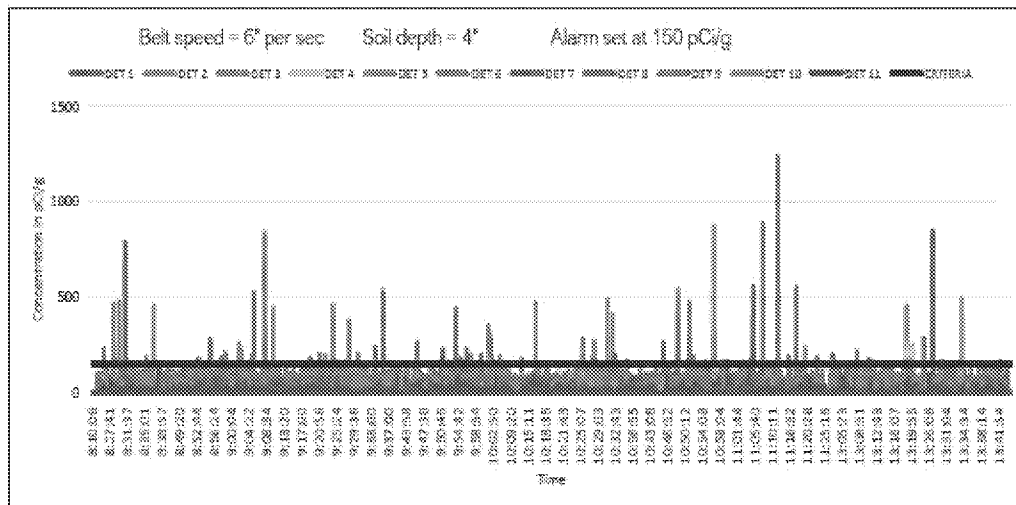
Figure 4-3. Plan view of a typical S3 footprint orientation



# S3 SOIL SORTING SYSTEM

## CALCULATED ACTIVITY FOR FILE

### EXAMPLE



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Figure 5-1. Example of the S3's Batch Chart Output

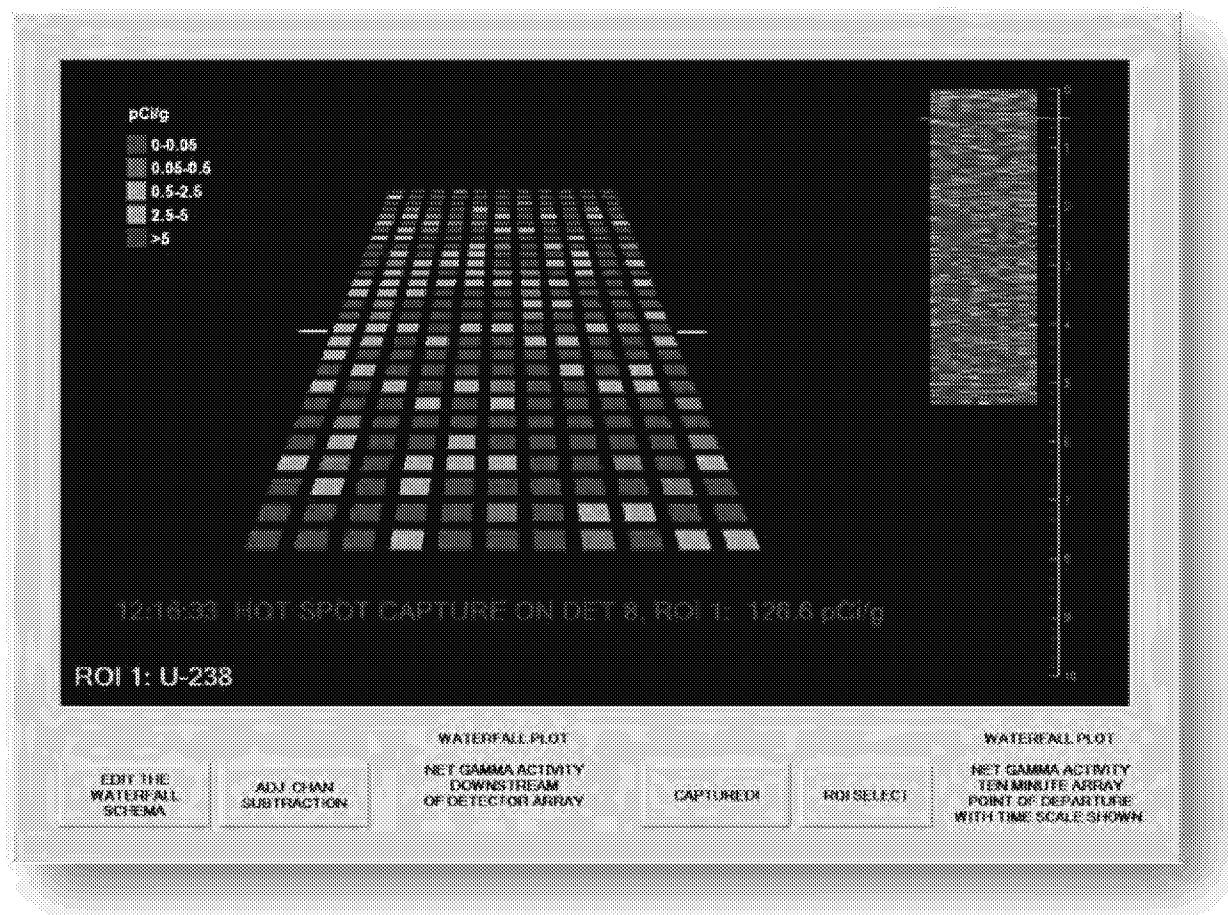


Figure 5-2. A Human-Machine Interface page in the S3 software highlighting The Software's Display Of A Found Source On The Belt Of The S3, Shown In Red

## Tables

**Table 1-1**  
**S3 NaI Detector Regions of Interest**

ROI	Span (keV)	Nuclide	Alarming	RG (pCi/g)***
ROI 1	10 -1100	Ra progeny gamma	Yes	1.0 + Bkgd.
ROI 2	540 - 670	<sup>214</sup> Bi 609 keV peak	Yes	1.0
ROI 3	625 - 698	<sup>137</sup> Cs 661.7 keV peak	Yes <sup>φ</sup>	0.113
ROI 4	10 - 300	<sup>90</sup> Sr secondary x-rays	Conditional*	0.331
ROI 5	820 – 1060	<sup>228</sup> Ac 911 & 969 keV peaks	No**	1.69
ROI 6	2400 – 2750	<sup>208</sup> Tl 2615 keV peak	No**	1.69

\* If the Navy makes <sup>90</sup>Sr LLRO available, ISO will perform field validation in the current configuration to verify consistent sorting of <sup>90</sup>Sr objects using bremsstrahlung effects as a diversionary marker.

\*\* For Parcel E, ROIs 5 and 6 are being collected for informational purposes.

\*\*\* The RG for <sup>226</sup>Ra is in addition to background. For other ROCs, the action level is the RG or background, whichever value is higher. Reference background soil concentrations (in pCi/g) will be provided by the Navy and documented.

<sup>φ</sup> Compliance with the <sup>137</sup>Cs and <sup>90</sup>Sr RGs will be based on the analytical data from soil sampling. These ROCs are treated identical to chemical contaminants in that final release decisions are based on the results of the sampling and analysis and are not based on field detection of elevated activity.

**Table 3-1****Instrument % Efficiency Values for Specified Regions of Interest under a Range of S3 Operating Parameters**

<b>Soil thickness (inch)</b>	<b>2</b>	<b>2</b>	<b>2.5</b>	<b>2.5</b>	<b>3</b>	<b>3</b>
<b>Soil Density (g/cc)</b>	<b>0.9</b>	<b>1.6</b>	<b>0.9</b>	<b>1.6</b>	<b>0.9</b>	<b>1.6</b>
LLRO <sup>226</sup> Ra (ROI 1)*	3.74	3.44	3.16	3.04	2.68	2.34
Diffuse <sup>226</sup> Ra (ROI 1)*	5.51	4.96	5.28	4.65	5.08	4.37
Diffuse <sup>137</sup> Cs (ROI 3)	1.83	1.47	1.69	1.32	1.57	1.19

\*assuming secular equilibrium between <sup>226</sup>Ra and <sup>214</sup>Pb/Bi

**Table 3-2****Minimum Detectable Concentrations (pCi/g) for Diffuse  $^{226}\text{Ra}$  and  $^{137}\text{Cs}$** 

<b>Belt Speed (/sec)</b>	<b>Soil thickness</b>	<b>2"</b>		<b>2.5"</b>		<b>3"</b>	
	<b>Soil Density (g/cc)</b>	<b>0.9</b>	<b>1.6</b>	<b>0.9</b>	<b>1.6</b>	<b>0.9</b>	<b>1.6</b>
7"	$^{226}\text{Ra}$ [10 keV - 1110 keV] *	0.1458	0.1213	0.1360	0.1157	0.1290	0.1124
7"	$^{137}\text{Cs}$ [625 keV - 698 keV]	0.0757	0.0702	0.0793	0.0700	0.0716	0.0706

\*assuming secular equilibrium between  $^{226}\text{Ra}$  and  $^{214}\text{Pb/Bi}$

**Table 3-3**  
**Minimum Detectable Activity (μCi) for a <sup>226</sup>Ra LLRO**

<b>Belt Speed (/sec)</b>	<b>Soil thickness</b>	<b>2"</b>		<b>2.5"</b>		<b>3"</b>	
	<b>Soil Density (g/cc)</b>	<b>0.9</b>	<b>1.6</b>	<b>0.9</b>	<b>1.6</b>	<b>0.9</b>	<b>1.6</b>
7"	<sup>226</sup> Ra [10 keV - 1110 keV] *	0.018	0.026	0.024	0.032	0.030	0.046

\*assuming secular equilibrium between <sup>226</sup>Ra and <sup>214</sup>Pb/Bi

**Table 3-4**  
**Equilibrium-Modified Instrument % Efficiency Values and Minimum Detectable Concentrations for Diffuse  $^{226}\text{Ra}$  (ROI 1) under a Range of Operating Parameters\***

Soil Thickness (inches)	Soil Density (g/cc)	Equilibrium factor ( $^{214}\text{Pb}$ : $^{226}\text{Ra}$ )	Instrument % Efficiency	MDC (pCi/g)
2	0.9	1	5.51 E-02	0.1458
		0.9	4.96 E-02	0.1620
		0.8	4.40 E-02	0.1823
		0.7	3.86 E-02	0.2083
		0.6	3.30 E-02	0.2430
		0.5	2.75 E-02	0.2916
	1.6	1	4.96 E-02	0.1213
		0.9	4.46 E-02	0.1348
		0.8	3.97 E-02	0.1516
		0.7	3.47 E-02	0.1733
		0.6	2.97 E-02	0.2022
		0.5	2.48 E-02	0.2426
2.5	0.9	1	5.28 E-02	0.1360
		0.9	4.75 E-02	0.1511
		0.8	4.22 E-02	0.1700
		0.7	3.70 E-02	0.1943
		0.6	3.17 E-02	0.2267
		0.5	2.64 E-02	0.2720
	1.6	1	4.65 E-02	0.1157
		0.9	4.19 E-02	0.1286
		0.8	3.72 E-02	0.1446
		0.7	3.26 E-02	0.1653
		0.6	2.79 E-02	0.1928
		0.5	2.33 E-02	0.2314
3	0.9	1	5.08 E-02	0.1290
		0.9	4.57 E-02	0.1433
		0.8	4.06 E-02	0.1613
		0.7	3.55 E-02	0.1843
		0.6	3.05 E-02	0.2150
		0.5	2.54 E-02	0.2580
	1.6	1	4.37 E-02	0.1124
		0.9	3.93 E-02	0.1249
		0.8	3.50 E-02	0.1405
		0.7	3.06 E-02	0.1606
		0.6	2.62 E-02	0.1873
		0.5	2.19 E-02	0.2248

\*Calculated MDCs based on 7" per second belt speed



**Table 3-5**  
**Equilibrium-Modified Instrument % Efficiency Values and Minimum Detectable Concentrations for Diffuse  $^{232}\text{Th}$  ( $^{228}\text{Ac}$ , ROI 5) under a Range of Operating Parameters\***

Soil Thickness (inches)	Soil Density (g/cc)	Equilibrium factor	Instrument % Efficiency	MDC (pCi/g)
2	1.6	1	3.49 E-03	0.27
		0.9	3.42 E-03	0.27
		0.8	3.36 E-03	0.28
		0.7	3.29 E-03	0.29
		0.6	3.23 E-03	0.29
		0.5	3.16 E-03	0.30
3	1.6	1	2.94 E-03	0.24
		0.9	2.88 E-03	0.24
		0.8	2.82 E-03	0.25
		0.7	2.76 E-03	0.25
		0.6	2.70 E-03	0.26
		0.5	2.65 E-03	0.26

\*Calculated MDCs based on 6" per second belt speed

**Table 3-6**  
**Equilibrium-Modified Instrument % Efficiency Values and Minimum Detectable Concentrations for Diffuse  $^{232}\text{Th}$  ( $^{208}\text{Tl}$ , ROI 6) under a Range of Operating Parameters\***

Soil Thickness (inches)	Soil Density (g/cc)	Equilibrium factor	Instrument % Efficiency	MDC (pCi/g)
2	1.6	1	1.63 E-03	0.22
		0.9	1.47 E-03	0.25
		0.8	1.30 E-03	0.28
		0.7	1.14 E-03	0.32
		0.6	9.77 E-04	0.37
		0.5	8.14 E-04	0.44
3	1.6	1	1.40 E-03	0.19
		0.9	1.26 E-03	0.21
		0.8	1.12 E-03	0.23
		0.7	9.83 E-04	0.27
		0.6	8.42 E-04	0.31
		0.5	7.02 E-04	0.37

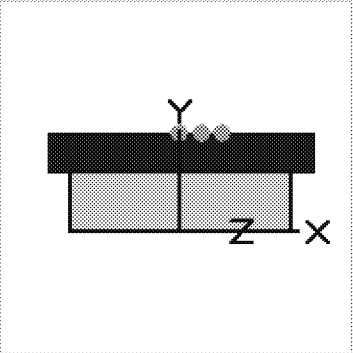
\*Calculated MDCs based on 6" per second belt speed

## **Appendix G**

# **Gamma Scan Minimum Detectable Concentration Calculations**

3x3 NaI  
Ra-226 Calculation

MicroShield 8.02 CB&I Federal Services (8.02-0000)				
Date	By	Checked		
Filename	Run Date	Run Time	Duration	
TI1AD 20141104.msdc	November 4, 2014	1:41:28 PM	00:00:00	
Project Info				
Case Title	TI1AD			
Description	3x3 Soil Distributed Source, no cover			
Geometry	8 - Cylinder Volume - End Shields			
Source Dimensions				
Height	15.0 cm (5.9 in)			
Radius	28.0 cm (11.0 in)			
Dose Points				
A	X	Y	Z	
#1	0.0 cm (0 in)	25.051 cm (9.9 in)	0.0 cm (0 in)	
#2	0.0 cm (0 in)	25.051 cm (9.9 in)	15.24 cm (6.0 in)	
#3	0.0 cm (0 in)	25.051 cm (9.9 in)	30.48 cm (1 ft)	
Shields				
Shield N	Dimension	Material	Density	
Source	3.69e+04 cm <sup>3</sup>	FGR 12 Soil	1.6	
Shield 1	10.0 cm	Air	0.00122	
Shield 2	.051 cm	Aluminum	2.7	
Air Gap		Air	0.00122	
Immersion		Air	0.00122	
Source Input: Grouping Method - Standard Indices				
Number of Groups: 25				
Lower Energy Cutoff: 0.015				
Photons < 0.015: Included				
Library: Grove				
Nuclide	Ci	Bq	μCi/cm <sup>3</sup>	Bq/cm <sup>3</sup>
Bi-210	3.5579e-008	1.3164e+003	9.6302e-007	3.5632e-002
Bi-214	5.8338e-008	2.1585e+003	1.5790e-006	5.8424e-002
Pb-210	3.5593e-008	1.3169e+003	9.6340e-007	3.5646e-002
Pb-214	5.8338e-008	2.1585e+003	1.5790e-006	5.8424e-002
Po-210	3.5185e-008	1.3018e+003	9.5235e-007	3.5237e-002
Po-214	5.8325e-008	2.1580e+003	1.5787e-006	5.8412e-002
Po-218	5.8349e-008	2.1589e+003	1.5794e-006	5.8436e-002
Ra-226	5.8349e-008	2.1589e+003	1.5793e-006	5.8436e-002
Rn-222	5.8349e-008	2.1589e+003	1.5794e-006	5.8436e-002



<b>Buildup: The material reference is Source</b>	
<b>Integration Parameters</b>	
Radial	20
Circumferential	10
Y Direction (axial)	10

<b>Results - Dose Point # 1 - (0,25.051,0) cm</b>					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate	Fluence Rate	Exposure Rate	Exposure Rate
		MeV/cm <sup>2</sup> /sec No Buildup	MeV/cm <sup>2</sup> /sec With Buildup	mR/hr No Buildup	mR/hr With Buildup
0.015	6.404e+02	1.678e-06	1.849e-06	1.439e-07	1.586e-07
0.05	7.720e+01	6.054e-05	1.556e-04	1.613e-07	4.145e-07
0.08	4.976e+02	1.047e-03	3.755e-03	1.657e-06	5.942e-06
0.1	2.930e+00	8.776e-06	3.221e-05	1.343e-08	4.928e-08
0.2	2.325e+02	1.795e-03	5.354e-03	3.168e-06	9.450e-06
0.3	4.454e+02	5.845e-03	1.474e-02	1.109e-05	2.796e-05
0.4	8.260e+02	1.581e-02	3.550e-02	3.081e-05	6.916e-05
0.5	3.856e+01	9.896e-04	2.037e-03	1.942e-06	3.999e-06
0.6	1.041e+03	3.393e-02	6.533e-02	6.624e-05	1.275e-04
0.8	2.040e+02	9.701e-03	1.691e-02	1.845e-05	3.216e-05
1.0	6.758e+02	4.303e-02	7.017e-02	7.932e-05	1.293e-04
1.5	4.109e+02	4.425e-02	6.464e-02	7.445e-05	1.088e-04
2.0	5.777e+02	8.959e-02	1.230e-01	1.385e-04	1.902e-04
<b>Totals</b>	<b>5.670e+03</b>	<b>2.461e-01</b>	<b>4.016e-01</b>	<b>4.260e-04</b>	<b>7.051e-04</b>

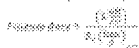
<b>Results - Dose Point # 2 - (0,25.051,15.24) cm</b>					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate	Fluence Rate	Exposure Rate	Exposure Rate
		MeV/cm <sup>2</sup> /sec No Buildup	MeV/cm <sup>2</sup> /sec With Buildup	mR/hr No Buildup	mR/hr With Buildup
0.015	6.404e+02	7.995e-07	9.012e-07	6.858e-08	7.730e-08
0.05	7.720e+01	5.352e-05	1.362e-04	1.426e-07	3.629e-07
0.08	4.976e+02	9.173e-04	3.240e-03	1.452e-06	5.127e-06
0.1	2.930e+00	7.672e-06	2.772e-05	1.174e-08	4.240e-08
0.2	2.325e+02	1.562e-03	4.615e-03	2.757e-06	8.145e-06
0.3	4.454e+02	5.078e-03	1.273e-02	9.633e-06	2.414e-05
0.4	8.260e+02	1.372e-02	3.070e-02	2.674e-05	5.982e-05
0.5	3.856e+01	8.583e-04	1.764e-03	1.685e-06	3.462e-06
0.6	1.041e+03	2.942e-02	5.661e-02	5.743e-05	1.105e-04
0.8	2.040e+02	8.408e-03	1.467e-02	1.599e-05	2.791e-05
1.0	6.758e+02	3.730e-02	6.095e-02	6.875e-05	1.123e-04
1.5	4.109e+02	3.837e-02	5.624e-02	6.456e-05	9.463e-05
2.0	5.777e+02	7.774e-02	1.072e-01	1.202e-04	1.657e-04
<b>Totals</b>	<b>5.670e+03</b>	<b>2.134e-01</b>	<b>3.489e-01</b>	<b>3.694e-04</b>	<b>6.123e-04</b>

<b>Results - Dose Point # 3 - (0,25.051,30.48) cm</b>					
Energy (MeV)	Activity (Photons/sec)				

Case Summary of TI1AD

		Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	6.404e+02	2.264e-07	2.562e-07	1.942e-08	2.198e-08
0.05	7.720e+01	2.547e-05	6.647e-05	6.786e-08	1.771e-07
0.08	4.976e+02	4.476e-04	1.658e-03	7.083e-07	2.623e-06
0.1	2.930e+00	3.772e-06	1.446e-05	5.771e-09	2.212e-08
0.2	2.325e+02	7.819e-04	2.498e-03	1.380e-06	4.409e-06
0.3	4.454e+02	2.569e-03	6.979e-03	4.874e-06	1.324e-05
0.4	8.260e+02	7.005e-03	1.696e-02	1.365e-05	3.305e-05
0.5	3.856e+01	4.414e-04	9.797e-04	8.664e-07	1.923e-06
0.6	1.041e+03	1.523e-02	3.157e-02	2.973e-05	6.162e-05
0.8	2.040e+02	4.401e-03	8.237e-03	8.371e-06	1.567e-05
1.0	6.758e+02	1.971e-02	3.438e-02	3.632e-05	6.337e-05
1.5	4.109e+02	2.064e-02	3.204e-02	3.473e-05	5.390e-05
2.0	5.777e+02	4.236e-02	6.148e-02	6.551e-05	9.507e-05
<b>Totals</b>	<b>5.670e+03</b>	<b>1.136e-01</b>	<b>1.969e-01</b>	<b>1.962e-04</b>	<b>3.451e-04</b>

3x3 NaI Scan for Ra-226 @ 1pCi/g, 0 inches of soil cover, 15 cm thick x 28 cm Radius, 0" offset  
Fluence rate to exposure rate (FRER, no units) =  $\sim (1 \text{ uR/h})(E\gamma)(u_{\text{en}}/\rho_{\text{air}})$



Mass energy-absorption coefficients from 1970 Rad Health Handbook

TABLE 1		
Energy, keV	$(u_{\text{en}}/\rho)_{\text{air}}$ , cm <sup>2</sup> /g	FRER
15	1.29	0.0517
20	0.516	0.0969
30	0.147	0.2288
40	0.094	0.3906
50	0.0584	0.5208
60	0.0292	0.5708
80	0.0236	0.5297
100	0.0231	0.4329
150	0.0251	0.2856
200	0.0288	0.1868
300	0.0288	0.1157
400	0.0296	0.0845
500	0.0297	0.0673
600	0.0296	0.0563
800	0.0289	0.0433
1,000	0.0280	0.0367
1,500	0.0255	0.0281
2,000	0.0234	0.0214

Probability of interaction (P) through end of detector given energy is

For Ludlum 3x3 Model 44-20: 7.6 cm dia x 7.6 cm thick NaI crystal

$x = 7.6$  cm

Aluminum housing for 44-20 is 0.020 inches (0.051 cm)

$\rho_{\text{NaI}} = 3.67$  g/cm<sup>3</sup>

Mass attenuation coefficients from 1970 Rad Health Handbook

TABLE 2		
Energy, keV	$(\mu/\rho)_{\text{NaI}}$ , cm <sup>2</sup> /g	P
15	47.4	1.00
20	22.3	1.00
30	7.45	1.00
40	19.3	1.00
50	10.7	1.00
60	6.62	1.00
80	3.12	1.00
100	1.72	1.00
150	0.825	1.00
200	0.334	1.00
300	0.167	0.99
400	0.117	0.96
500	0.0955	0.93
600	0.0826	0.90
800	0.0676	0.85
1,000	0.0568	0.80
1,500	0.0469	0.73
2,000	0.0413	0.68

Relative Detector Response (RDR) = relative fluence-to-exposure rate (FRER) times probability (P) of interaction

Relative Detector Response = Fluence Rate x Probability of Interaction

TABLE 3			
Energy, keV	Fluence Rate	P	RDR
15	0.0517	1.00	0.0517
20	0.0969	1.00	0.0969
30	0.2288	1.00	0.2288
40	0.3906	1.00	0.3906
50	0.5208	1.00	0.5208
60	0.5708	1.00	0.5708
80	0.5297	1.00	0.5297
100	0.4329	1.00	0.4329
150	0.2856	1.00	0.2856
200	0.1868	1.00	0.1868
300	0.1157	0.99	0.1146
400	0.0845	0.96	0.0812
500	0.0673	0.93	0.0626
600	0.0563	0.90	0.0507
800	0.0433	0.85	0.0367
1,000	0.0367	0.80	0.0287
1,500	0.0281	0.73	0.0191
2,000	0.0214	0.68	0.0146

Estimated Ludlum 44-20 7.6 cm dia x 7.6 cm thick NaI response for Cs-137 is 2300 cpm/ $\mu\text{R/hr}$

Use same methodology and interpolating for Cs-137 response have:

Energy, keV	$(u_{\text{en}}/\rho)_{\text{air}}$ , cm <sup>2</sup> /g	FRER =	0.0514
662	0.0294		
Energy, keV	$(\mu/\rho)_{\text{NaI}}$ , cm <sup>2</sup> /g	Probability =	0.89
662	0.0780		
		RDR =	0.0455

For this detector the response to another energy is based on the ratio of the relative detector response, RDR, to the Cs-137 energy

Relative Detector Response to another energy =  $\frac{\text{RDR}_{\text{other}}}{\text{RDR}_{\text{Cs-137}}} \times \text{Response}_{\text{Cs-137}}$

TABLE 4		
Energy, keV	RDR <sub>662</sub>	Ludlum 44-20 3x3 NaI cpm per $\mu\text{R/hr}$ , E
15	0.0517	2610
20	0.0969	4894
30	0.2288	15453
40	0.3906	19729
50	0.5208	26306
60	0.5708	28828
80	0.5297	26752
100	0.4329	21865
150	0.2856	13415
200	0.1868	8422
300	0.1146	5790
400	0.0812	4103
500	0.0626	3164
600	0.0507	2500
662	0.0455	2300
800	0.0367	1853
1,000	0.0287	1452
1,500	0.0191	964
2,000	0.0146	738

FRER = 11840 3341104

#### Assumptions

Detector is 3 in (7.6 cm) height by 3 in (7.6 cm) diameter

15 x 28 source at 1.6 g/cm<sup>3</sup> (dry soil) - Buildup reference

No soil cover

0.020 inch (0.051 cm) aluminum detector casing @ 2.7 g/cm<sup>3</sup>

2300 cpm/ $\mu\text{R/hr}$

Assume average background count rate of 18,000 cpm based on observed soil reference area data

Centerpoint of detector has no offset from center of source



MDCR<sub>surveyor</sub> for Cs-137 energy

2300 cpm/μR/hr  
Bkgd = 18000 cpm  
Observation Interval, i = 1 second  
b<sub>i</sub> = 300 counts  
d' = 1.38 from Table 6.1 of NUREG-1507  
MDCR = 1434.14 cpm  
MDCR<sub>sample</sub> = 2028 cpm  
minimum detectable exposure rate, Cs-137 = 0.88 μR/hr

Table 5				
Energy, keV	Microshield Exposure Rate, μR/hr (with buildup)	cpm/μR/hr	cpm/μR/hr (weighted)	Percent of total detector response
15	1.159E-04	2810	0	0.0%
20	0.000E+00	4894	0	0.0%
30	0.000E+00	11463	0	0.0%
40	0.000E+00	19729	0	0.0%
50	2.393E-04	29308	9	0.4%
60	0.000E+00	28828	0	0.0%
80	3.929E-03	28762	152	7.3%
100	3.551E-05	21885	1	0.1%
150	0.000E+00	13415	0	0.0%
200	8.324E-03	8422	114	5.5%
300	2.610E-02	6790	219	10.5%
400	6.605E-02	4103	392	18.9%
500	3.978E-03	2164	18	0.9%
600	1.244E-01	250	481	22.2%
800	3.170E-02	1853	85	4.1%
1000	1.280E-01	1452	269	13.0%
1500	1.082E-01	854	151	7.3%
2000	1.899E-01	738	203	9.8%
Total	6.909E-01		2074	100.0%

$$S_i = d' \sqrt{b_i}$$

$$MDCR = S_i (60 / i)$$

$$MDCR_{SURVEYOR} = \frac{MDCR}{\sqrt{P}}$$

P=0.50

mR/hr, from MS	
15	1.15E-07
20	
30	
40	
50	2.39E-07
60	
80	3.93E-06
100	3.55E-08
150	
200	8.32E-06
300	2.61E-05
400	6.61E-05
500	3.98E-06
600	1.24E-04
800	3.17E-05
1000	1.28E-04
1500	1.08E-04
2000	1.90E-04
	6.909E-04

Microshield Output					
0.015	6.40E+02	1.68E-05	1.85E-06	1.44E-07	1.59E-07
0.05	7.72E+01	6.05E-05	1.56E-04	1.61E-07	4.15E-07
0.08	4.98E+02	1.05E-03	3.76E-03	1.66E-06	5.94E-06
0.1	2.93E+00	8.78E-06	3.22E-05	1.34E-08	4.93E-08
0.2	2.33E+02	1.80E-03	5.35E-03	3.17E-06	9.45E-06
0.3	4.45E+02	5.85E-03	1.47E-02	1.11E-05	2.80E-05
0.4	8.26E+02	1.59E-02	3.55E-02	3.06E-05	6.92E-05
0.5	3.86E+01	9.80E-04	2.04E-02	1.54E-06	4.00E-06
0.6	1.04E+03	3.39E-02	6.53E-02	6.62E-05	1.28E-04
0.8	2.04E+02	9.70E-03	1.69E-02	1.85E-05	3.22E-05
1	6.76E+02	4.30E-02	7.02E-02	7.93E-05	1.29E-04
1.5	4.11E+02	4.43E-02	6.46E-02	7.45E-05	1.09E-04
2	5.78E+02	8.86E-02	1.23E-01	1.33E-04	1.90E-04

Minimum Detectable Exposure Rate =

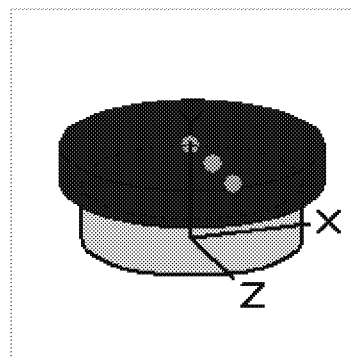
$$MDCR_{surveyor}(\text{cpm}/\mu\text{R/hr}) = \frac{2.33 \times 10^2 \times 60}{0.978} = 1434.14 \text{ cpm}/\mu\text{R/hr}$$

and MDC for Ra-228 based on a normalized 1 pCi/g in soil with density of 1.6 g/cm<sup>3</sup>

$$\text{Scan MDC} = \frac{1.43}{5.24E+01} = 2.73E-02 \text{ pCi/g}$$

3x3 NaI  
Cs-137 Calculation

MicroShield 8.02 CB&I Federal Services (8.02-0000)				
Date		By		Checked
Filename		Run Date	Run Time	Duration
TI1AD 20141104.msdl		March 12, 2019	1:29:36 PM	00:00:00
Project Info				
Case Title		TI1AD_Cs		
Description		3x3 Soil Distributed Source, no cover		
Geometry		8 - Cylinder Volume - End Shields		
Source Dimensions				
Height		15.0 cm (5.9 in)		
Radius		28.0 cm (11.0 in)		
Dose Points				
A	X	Y	Z	
#1	0.0 cm (0 in)	25.051 cm (9.9 in)	0.0 cm (0 in)	
#2	0.0 cm (0 in)	25.051 cm (9.9 in)	15.24 cm (6.0 in)	
#3	0.0 cm (0 in)	25.051 cm (9.9 in)	30.48 cm (1 ft)	
Shields				
Shield N	Dimension	Material	Density	
Source	3.69e+04 cm <sup>3</sup>	Soil_FGR12	1.6	
Shield 1	10.0 cm	Air	0.00122	
Shield 2	.051 cm	Aluminum	2.7	
Air Gap		Air	0.00122	
Immersion		Air	0.00122	
<b>Source Input: Grouping Method - Standard Indices</b> <b>Number of Groups: 25</b> <b>Lower Energy Cutoff: 0.015</b> <b>Photons &lt; 0.015: Included</b> <b>Library: Grove</b>				
Nuclide	Ci	Bq	μCi/cm <sup>3</sup>	Bq/cm <sup>3</sup>
Ba-137m	5.9112e-008	2.1872e+003	1.6000e-006	5.9200e-002
Cs-137	5.5920e-008	2.0690e+003	1.5136e-006	5.6003e-002
Buildup: The material reference is Source				
Integration Parameters				
Radial				20
Circumferential				10
Y Direction (axial)				10
Results - Dose Point # 1 - (0,25.051,0) cm				
Energy (MeV)	Activity (Photons/sec)			



		Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	2.270e+01	5.947e-08	6.554e-08	5.101e-09	5.622e-09
0.03	1.288e+02	1.851e-05	2.766e-05	1.834e-07	2.741e-07
0.04	3.040e+01	1.245e-05	2.409e-05	5.507e-08	1.065e-07
0.6	1.968e+03	6.417e-02	1.235e-01	1.253e-04	2.411e-04
<b>Totals</b>	<b>2.150e+03</b>	<b>6.420e-02</b>	<b>1.236e-01</b>	<b>1.255e-04</b>	<b>2.415e-04</b>

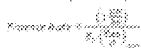
**Results - Dose Point # 2 - (0,25.051,15.24) cm**

Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	2.270e+01	2.835e-08	3.195e-08	2.431e-09	2.741e-09
0.03	1.288e+02	1.652e-05	2.487e-05	1.637e-07	2.464e-07
0.04	3.040e+01	1.107e-05	2.131e-05	4.895e-08	9.427e-08
0.6	1.968e+03	5.564e-02	1.070e-01	1.086e-04	2.089e-04
<b>Totals</b>	<b>2.150e+03</b>	<b>5.566e-02</b>	<b>1.071e-01</b>	<b>1.088e-04</b>	<b>2.093e-04</b>

**Results - Dose Point # 3 - (0,25.051,30.48) cm**

Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	2.270e+01	8.025e-09	9.084e-09	6.883e-10	7.792e-10
0.03	1.288e+02	7.397e-06	1.135e-05	7.331e-08	1.125e-07
0.04	3.040e+01	5.172e-06	1.017e-05	2.287e-08	4.496e-08
0.6	1.968e+03	2.880e-02	5.970e-02	5.622e-05	1.165e-04
<b>Totals</b>	<b>2.150e+03</b>	<b>2.881e-02</b>	<b>5.972e-02</b>	<b>5.631e-05</b>	<b>1.167e-04</b>

3x3 NaI Scan for Cs-137 @ 1pCi/g, 0 inches of soil cover, 15 cm thick x 28 cm Radius, 0" offset  
Fluence rate to exposure rate (FRER, no units) =  $\sim (1 \text{ uR/h})(E\gamma)(u_{\text{eff}}/\rho)$



Mass energy-absorption coefficients from 1970 Rad Health Handbook

Energy, keV	TABLE 1 ( $u_{\text{eff}}/\rho_{\text{NaI}}$ , cm <sup>2</sup> /g)	FRER
15	1.29	0.0517
20	0.516	0.0969
30	0.147	0.2288
40	0.094	0.3906
50	0.0584	0.5208
60	0.0292	0.5708
80	0.0236	0.5297
100	0.0231	0.4329
150	0.0251	0.2856
200	0.0288	0.1868
300	0.0288	0.1157
400	0.0296	0.0845
500	0.0297	0.0673
600	0.0296	0.0563
800	0.0289	0.0433
1,000	0.0280	0.0367
1,500	0.0255	0.0281
2,000	0.0234	0.0214

Probability of interaction (P) through end of detector for given energy is

For Ludlum 3x3 Model 44-20: 7.6 cm dia x 7.6 cm thick NaI crystal

$x =$

$\rho_{\text{NaI}} = 3.67 \text{ g/cm}^3$

Aluminum housing for 44-20 is 0.020 inches (0.051 cm)

Mass attenuation coefficients from 1970 Rad Health Handbook

Energy, keV	TABLE 2 ( $\mu/\rho_{\text{NaI}}$ , cm <sup>2</sup> /g)	P
15	47.4	1.00
20	22.3	1.00
30	7.45	1.00
40	19.3	1.00
50	10.7	1.00
60	6.62	1.00
80	3.12	1.00
100	1.72	1.00
150	0.825	1.00
200	0.334	1.00
300	0.167	0.99
400	0.117	0.96
500	0.0955	0.93
600	0.0826	0.90
800	0.0676	0.85
1,000	0.0568	0.80
1,500	0.0469	0.73
2,000	0.0413	0.68

Relative Detector Response (RDR) = relative fluence-to-exposure rate (FRER) times probability (P) of interaction

Estimated Ludlum 3x3 Model 44-20 NaI response for Cs-137 is

Energy, keV	Fluence Rate	P	RDR
15	0.0517	1.00	0.0517
20	0.0969	1.00	0.0969
30	0.2288	1.00	0.2288
40	0.3906	1.00	0.3906
50	0.5208	1.00	0.5208
60	0.5708	1.00	0.5708
80	0.5297	1.00	0.5297
100	0.4329	1.00	0.4329
150	0.2856	1.00	0.2856
200	0.1868	1.00	0.1868
300	0.1157	0.99	0.1146
400	0.0845	0.96	0.0812
500	0.0673	0.93	0.0626
600	0.0563	0.90	0.0507
800	0.0433	0.85	0.0367
1,000	0.0367	0.80	0.0287
1,500	0.0281	0.73	0.0191
2,000	0.0214	0.68	0.0146

Estimated Ludlum 44-20 7.6 cm dia x 7.6 cm thick NaI response for Cs-137 is 2300 cpm/ $\mu\text{R/hr}$

Use same methodology and interpolating for Cs-137 response have:

Energy, keV	( $u_{\text{eff}}/\rho_{\text{NaI}}$ , cm <sup>2</sup> /g)	FRER =	0.0514
662	0.0294		
Energy, keV	( $\mu/\rho_{\text{NaI}}$ , cm <sup>2</sup> /g)	Probability =	0.89
662	0.0780		
		RDR =	0.0455

For this detector the response to another energy is based on the ratio of the relative detector response, RDR, to the Cs-137 energy

Estimated Ludlum 3x3 Model 44-20 NaI response for Cs-137 is

Energy, keV	RDR <sub>662</sub>	Ludlum 44-20 3x3 NaI cpm per $\mu\text{R/hr}$ , E
15	0.0517	2610
20	0.0969	4894
30	0.2288	15453
40	0.3906	19729
50	0.5208	26306
60	0.5708	28828
80	0.5297	26752
100	0.4329	21865
150	0.2856	13415
200	0.1868	8422
300	0.1146	5790
400	0.0812	4103
500	0.0626	3164
600	0.0507	2560
662	0.0455	2300
800	0.0367	1853
1,000	0.0287	1452
1,500	0.0191	964
2,000	0.0146	738

FRER = 11840.28

Assumptions

Detector is 3 in (7.6 cm) height by 3 in (7.6 cm) diameter

15 x 28 source at 1.6 g/cm<sup>3</sup> (dry soil) - Buildup reference

No soil cover

0.020 inch (0.051 cm) aluminum detector casing @ 2.7 g/cm<sup>3</sup>  
2300 cpm/ $\mu\text{R/hr}$

Assume average background count rate of 18,000 cpm based on observed soil reference area data

Centerpoint of detector has no offset from center of source

MDCR<sub>surveyor</sub> for Cs-137 energy

2300 cpm/μR/hr  
Bgkd = 18000 cpm  
Observation Interval, i = 1 second  
b<sub>i</sub> = 300 counts  
d' = 1.38 from Table 8.1 of NUREG-1507  
MDCR = 1434.14 cpm  
MDCR<sub>surveyor</sub> = 2028 cpm  
minimum detectable exposure rate, Cs-137 = 0.88 μR/hr

Table 5				
Energy, keV	Microshield Exposure Rate, μR/hr (with buildup)	cpm/μR/hr	cpm/μR/hr (weighted)	Percent of total detector response
15	5.62E-06	2810	0	0.0%
20	0.00E+00	4894	0	0.0%
30	2.74E-04	11463	13	0.5%
40	1.06E-04	19729	9	0.3%
50	0.00E+00	29308	0	0.0%
60	0.00E+00	28828	0	0.0%
80	0.00E+00	28762	0	0.0%
100	0.00E+00	21885	0	0.0%
150	0.00E+00	13415	0	0.0%
200	0.00E+00	8422	0	0.0%
300	0.00E+00	6790	0	0.0%
400	0.00E+00	4103	0	0.0%
500	0.00E+00	2164	0	0.0%
600	2.41E-01	2500	2568	99.2%
800	0.00E+00	1853	0	0.0%
1000	0.00E+00	1452	0	0.0%
1500	0.00E+00	864	0	0.0%
2000	0.00E+00	738	0	0.0%
Total	2.41E-01		2578	100.0%

$$S_i = d' \sqrt{b_i}$$

$$MDCR = S_i (60 / i)$$

$$MDCR_{SURVEYOR} = \frac{MDCR}{\sqrt{P}}$$

P=0.50

mR/hr, from MS	
15	5.62E-06
20	
30	2.74E-07
40	1.07E-07
50	
60	
80	
100	
150	
200	
300	
400	
500	
600	2.41E-04
800	
1000	
1500	
2000	
2.415E-04	

Microshield Output					
0.015	2.27E+01	5.95E-08	5.55E-08	5.10E-09	5.62E-09
0.03	1.29E+02	1.85E-05	2.77E-05	1.83E-07	2.74E-07
0.04	3.04E+01	1.25E-05	2.41E-05	5.51E-08	1.07E-07
0.1					
0.2					
0.3					
0.4					
0.5					
0.6	1.37E+03	6.42E-02	1.24E-01	1.25E-04	2.41E-04
0.8					
1					
1.5					
2					

Minimum Detectable Exposure Rate =

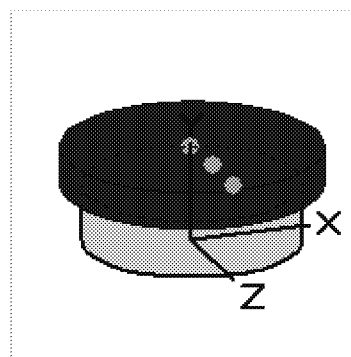
$$\frac{MDCR_{surveyor}(\text{cpm}/\mu\text{R/hr})}{0.787} = \frac{2028 \text{ cpm}/\mu\text{R/hr}}{0.787} = 2578 \text{ cpm}/\mu\text{R/hr}$$

and MDC for Ra-228 based on a normalized 1 pCi/g in soil with density of 1.6 g/cm<sup>3</sup>

$$\text{Scan MDC} = \frac{3.28}{1.21E+02} = \frac{3.28}{121} = 0.0271 \text{ pCi/g} = 0.0271 \text{ Bq/kg}$$

3x3 NaI  
Th-232 Calculation

MicroShield 8.02 Aptim (8.02-0000)				
Date		By		Checked
Filename		Run Date	Run Time	Duration
TI1AD 20141104.msd		September 3, 2019	2:50:23 PM	00:00:00
Project Info				
Case Title		TI1AD_Th		
Description		3x3 Soil Distributed Source, no cover		
Geometry		8 - Cylinder Volume - End Shields		
Source Dimensions				
Height		15.0 cm (5.9 in)		
Radius		28.0 cm (11.0 in)		
Dose Points				
A	X	Y	Z	
#1	0.0 cm (0 in)	25.051 cm (9.9 in)	0.0 cm (0 in)	
#2	0.0 cm (0 in)	25.051 cm (9.9 in)	15.24 cm (6.0 in)	
#3	0.0 cm (0 in)	25.051 cm (9.9 in)	30.48 cm (1 ft)	
Shields				
Shield N	Dimension	Material	Density	
Source	3.69e+04 cm³	FGR12_Soil	1.6	
Shield 1	10.0 cm	Air	0.00122	
Shield 2	.051 cm	Aluminum	2.7	
Air Gap		Air	0.00122	
Immersion		Air	0.00122	
Source Input: Grouping Method - Standard Indices				
Number of Groups: 25				
Lower Energy Cutoff: 0.015				
Photons < 0.015: Included				
Library: Grove				
Nuclide	Ci	Bq	µCi/cm³	Bq/cm³
Ac-228	5.9112e-008	2.1871e+003	1.6000e-006	5.9200e-002
Bi-212	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Pb-212	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Po-212	3.7873e-008	1.4013e+003	1.0251e-006	3.7929e-002
Po-216	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Ra-224	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Ra-228	5.9112e-008	2.1871e+003	1.6000e-006	5.9200e-002
Rn-220	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Th-228	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Th-232	5.9112e-008	2.1872e+003	1.6000e-006	5.9200e-002
Tl-208	2.1239e-008	7.8584e+002	5.7487e-007	2.1270e-002





Buildup: The material reference is Source					
Integration Parameters					
Radial					20
Circumferential					10
Y Direction (axial)					10
Results - Dose Point # 1 - (0,25.051,0) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	1.787e+03	4.681e-06	5.159e-06	4.015e-07	4.425e-07
0.04	2.236e+01	9.160e-06	1.772e-05	4.051e-08	7.836e-08
0.06	1.512e+01	1.832e-05	5.610e-05	3.639e-08	1.114e-07
0.08	9.439e+02	1.985e-03	7.122e-03	3.142e-06	1.127e-05
0.1	1.552e+02	4.649e-04	1.706e-03	7.112e-07	2.610e-06
0.15	9.193e+01	4.850e-04	1.623e-03	7.987e-07	2.672e-06
0.2	1.188e+03	9.170e-03	2.736e-02	1.619e-05	4.828e-05
0.3	5.702e+02	7.482e-03	1.887e-02	1.419e-05	3.579e-05
0.4	5.175e+01	9.906e-04	2.224e-03	1.930e-06	4.333e-06
0.5	3.034e+02	7.786e-03	1.603e-02	1.528e-05	3.146e-05
0.6	6.954e+02	2.268e-02	4.365e-02	4.426e-05	8.521e-05
0.8	6.969e+02	3.314e-02	5.777e-02	6.304e-05	1.099e-04
1.0	1.274e+03	8.110e-02	1.322e-01	1.495e-04	2.437e-04
1.5	2.934e+02	3.159e-02	4.615e-02	5.315e-05	7.764e-05
2.0	6.638e+00	1.029e-03	1.413e-03	1.592e-06	2.186e-06
3.0	7.843e+02	2.005e-01	2.559e-01	2.720e-04	3.472e-04
Totals	8.879e+03	3.984e-01	6.122e-01	6.362e-04	1.003e-03
Results - Dose Point # 2 - (0,25.051,15.24) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	1.787e+03	2.231e-06	2.515e-06	1.914e-07	2.157e-07
0.04	2.236e+01	8.141e-06	1.568e-05	3.601e-08	6.934e-08
0.06	1.512e+01	1.613e-05	4.884e-05	3.204e-08	9.702e-08
0.08	9.439e+02	1.740e-03	6.145e-03	2.753e-06	9.725e-06
0.1	1.552e+02	4.064e-04	1.468e-03	6.217e-07	2.246e-06
0.15	9.193e+01	4.228e-04	1.397e-03	6.962e-07	2.300e-06
0.2	1.188e+03	7.981e-03	2.358e-02	1.409e-05	4.162e-05
0.3	5.702e+02	6.501e-03	1.629e-02	1.233e-05	3.091e-05
0.4	5.175e+01	8.598e-04	1.923e-03	1.675e-06	3.748e-06
0.5	3.034e+02	6.753e-03	1.388e-02	1.325e-05	2.724e-05
0.6	6.954e+02	1.966e-02	3.783e-02	3.837e-05	7.383e-05
0.8	6.969e+02	2.872e-02	5.013e-02	5.464e-05	9.535e-05
1.0	1.274e+03	7.028e-02	1.149e-01	1.296e-04	2.117e-04
1.5	2.934e+02	2.739e-02	4.015e-02	4.608e-05	6.755e-05
2.0	6.638e+00	8.932e-04	1.231e-03	1.381e-06	1.904e-06

3.0	7.843e+02	1.742e-01	2.233e-01	2.363e-04	3.030e-04
<b>Totals</b>	<b>8.879e+03</b>	<b>3.458e-01</b>	<b>5.322e-01</b>	<b>5.520e-04</b>	<b>8.715e-04</b>

Results - Dose Point # 3 - (0,25.051,30.48) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	1.787e+03	6.317e-07	7.150e-07	5.418e-08	6.133e-08
0.04	2.236e+01	3.805e-06	7.478e-06	1.683e-08	3.307e-08
0.06	1.512e+01	7.767e-06	2.427e-05	1.543e-08	4.821e-08
0.08	9.439e+02	8.489e-04	3.144e-03	1.343e-06	4.975e-06
0.1	1.552e+02	1.998e-04	7.658e-04	3.057e-07	1.172e-06
0.15	9.193e+01	2.101e-04	7.472e-04	3.460e-07	1.230e-06
0.2	1.188e+03	3.995e-03	1.276e-02	7.051e-06	2.253e-05
0.3	5.702e+02	3.289e-03	8.934e-03	6.239e-06	1.695e-05
0.4	5.175e+01	4.389e-04	1.063e-03	8.551e-07	2.071e-06
0.5	3.034e+02	3.473e-03	7.708e-03	6.817e-06	1.513e-05
0.6	6.954e+02	1.018e-02	2.110e-02	1.986e-05	4.118e-05
0.8	6.969e+02	1.504e-02	2.814e-02	2.860e-05	5.353e-05
1.0	1.274e+03	3.714e-02	6.479e-02	6.845e-05	1.194e-04
1.5	2.934e+02	1.474e-02	2.287e-02	2.479e-05	3.848e-05
2.0	6.638e+00	4.868e-04	7.064e-04	7.527e-07	1.092e-06
3.0	7.843e+02	9.655e-02	1.292e-01	1.310e-04	1.753e-04
<b>Totals</b>	<b>8.879e+03</b>	<b>1.866e-01</b>	<b>3.020e-01</b>	<b>2.965e-04</b>	<b>4.932e-04</b>

3x3 NaI Scan for Th-232 @ 1pCi/g, 0 inches of soil cover, 15 cm thick x 28 cm Radius, 0" offset  
Fluence rate to exposure rate (FRER, no units) =  $\sim (1 \text{ uR/h})(E\gamma)(u_{\text{air}}/\rho)_{\text{air}}$

$$\text{Fluence Rate} = \frac{A}{4\pi r^2} \times \frac{1}{\rho_{\text{air}}} \times \frac{1}{E_{\gamma}} \times \frac{1}{\mu_{\text{air}}}$$

Mass energy-absorption coefficients from 1970 Rad Health Handbook

TABLE 1		
Energy, keV	$(u_{\text{air}}/\rho)_{\text{air}}$ , cm <sup>2</sup> /g	FRER
15	0.0517	0.0517
20	0.0516	0.0969
30	0.147	0.2288
40	0.064	0.3908
50	0.0584	0.5208
60	0.0292	0.5708
80	0.0236	0.5297
100	0.0251	0.4329
150	0.0251	0.2856
200	0.0288	0.1888
300	0.0288	0.1157
400	0.0286	0.0845
500	0.0297	0.0873
600	0.0286	0.0563
800	0.0289	0.0433
1,000	0.0280	0.0357
1,500	0.0255	0.0261
2,000	0.0234	0.0214
3,000	0.0205	0.0163

Probability of Interaction (P) through end of detector for given energy is

For Ludlum 3x3 Model 44-20: 7.6 cm dia x 7.6 cm thick NaI crystal  
Aluminum housing for 44-20 is 0.020 inches (0.051 cm)

$$x = 7.6 \text{ cm}$$

$$\rho_{\text{Al}} = 3.67 \text{ g/cm}^3$$

Mass attenuation coefficients from 1970 Rad Health Handbook

TABLE 2		
Energy, keV	$(\mu/\rho)_{\text{Al}}$ , cm <sup>2</sup> /g	P
15	47.4	1.00
20	22.3	1.00
30	7.45	1.00
40	19.3	1.00
50	10.7	1.00
60	6.82	1.00
80	3.12	1.00
100	1.72	1.00
150	0.625	1.00
200	0.334	1.00
300	0.167	0.99
400	0.117	0.96
500	0.0955	0.93
600	0.0826	0.90
800	0.0676	0.85
1,000	0.0586	0.80
1,500	0.0489	0.73
2,000	0.0413	0.68
3,000	0.0366	0.64

Relative Detector Response (RDR) = relative fluence-to-exposure rate (FRER) times probability (P) of interaction

Relative Detector Response = Fluence Rate x Probability of Interaction

TABLE 3			
Energy, keV	Fluence Rate	P	RDR
15	0.0517	1.00	0.0517
20	0.0969	1.00	0.0969
30	0.2288	1.00	0.2288
40	0.3908	1.00	0.3908
50	0.5208	1.00	0.5208
60	0.5708	1.00	0.5708
80	0.5297	1.00	0.5297
100	0.4329	1.00	0.4329
150	0.2856	1.00	0.2856
200	0.1888	1.00	0.1888
300	0.1157	0.99	0.1146
400	0.0845	0.96	0.0812
500	0.0873	0.93	0.0828
600	0.0563	0.90	0.0507
800	0.0433	0.85	0.0367
1,000	0.0357	0.80	0.0287
1,500	0.0261	0.73	0.0191
2,000	0.0214	0.68	0.0146
3,000	0.0163	0.64	0.0104

Estimated Ludlum 44-20 7.6 cm dia x 7.6 cm thick NaI response for Cs-137 is 2300 cpm/uR/hr

Use same methodology and interpolating for Cs-137 response have:

Energy, keV	$(u_{\text{air}}/\rho)_{\text{air}}$ , cm <sup>2</sup> /g	FRER	
662	0.0294	~	0.0514
Energy, keV	$(\mu/\rho)_{\text{Al}}$ , cm <sup>2</sup> /g	Probability	
662	0.0780	=	0.89
		RDR =	0.0455

For this detector the response to another energy is based on the ratio of the relative detector response, RDR, to the Cs-137 energy

$$\text{Response} = \frac{\text{RDR}_{\text{Cs-137}}}{\text{RDR}_{\text{Energy}}} \times \text{Response}_{\text{Cs-137}}$$

TABLE 4		
Energy, keV	RDR <sub>E</sub>	Ludlum 44-20 3x3 NaI cpm per uR/hr, E <sub>E</sub>
15	0.0517	2510
20	0.0969	4894
30	0.2288	11453
40	0.3908	19729
50	0.5208	26306
60	0.5708	28828
80	0.5297	26752
100	0.4329	21865
150	0.2856	13415
200	0.1888	9422
300	0.1146	5790
400	0.0812	4103
500	0.0828	3164
600	0.0507	2360
800	0.0455	2300
1,000	0.0367	1853
1,500	0.0287	1452
2,000	0.0191	964
3,000	0.0146	738
3,000	0.0104	525

File: TH40\_Th

#### Assumptions

Detector is 3 in (7.6 cm) height by 3 in (7.6 cm) diameter

15 x 28 source at 1.6 g/cm<sup>3</sup> (dry soil) - Buildup reference

No soil cover

0.020 in-th (0.051 cm) aluminum detector casing @ 2.7 g/cm<sup>3</sup>

2300 cpm/uR/hr

Assume average background count rate of 18,000 cpm based on observed soil reference area data

Centerpoint of detector has no offset from center of source

MDCR<sub>surveyor</sub> for Cs-137 energy

2300 cpm/μR/hr  
Bkgd = 18000 cpm  
Observation Interval, t = 1 second  
t<sub>0</sub> = 300 counts  
df = 1.38 from Table 6.1 of NUREG-1507  
MDCR = 1434.14 cpm  
MDCR<sub>surveyor</sub> = 2028 cpm

minimum detectable exposure rate, Cs-137 = 0.88 μR/hr

Energy, keV	MicroShield Exposure Rate, μR/hr (with buildup)	cpm/μR/hr	cpm/μR/hr (weighted)	Percent of NaI detector response
15	4.425E-04	2810	1	0.1%
20	0.000E+00	4894	0	0.0%
30	0.000E+00	11453	0	0.0%
40	7.836E-05	19729	2	0.1%
50	0.000E+00	26306	0	0.0%
60	1.144E-04	28628	3	0.1%
80	1.127E-02	26752	301	13.6%
100	2.610E-03	21865	57	2.6%
150	2.672E-03	13415	36	1.6%
200	4.628E-02	9422	454	20.5%
300	3.578E-02	5790	207	9.4%
400	4.333E-03	4103	18	0.8%
500	3.146E-02	3164	99	4.5%
600	8.521E-02	2560	217	9.9%
800	1.096E-01	1853	203	9.2%
1000	2.437E-01	1452	353	16.0%
1500	7.764E-02	964	75	3.4%
2000	2.186E-03	738	2	0.1%
3000	3.472E-01	525	182	8.2%
Total	1.003E+00	186423	2208	100.0%

$$S_i = d' \sqrt{b_i}$$

$$MDCR = S_i (60 / i)$$

$$MDCR_{SURVEYOR} = \frac{MDCR}{\sqrt{P}}$$

P=0.50

mR/hr, from MS	
15	4.43E-07
20	
30	
40	7.84E-08
50	
60	1.11E-07
80	1.13E-05
100	2.61E-06
150	2.67E-06
200	4.83E-05
300	3.58E-05
400	4.33E-06
500	3.15E-05
600	8.52E-05
800	1.10E-04
1000	2.44E-04
1500	7.76E-05
2000	2.19E-06
3000	3.47E-04
1.003E-03	

Microshield Output				
0.015	1.79E+03	4.68E-06	5.16E-06	4.02E-07
0.04	2.24E+01	9.16E-06	1.77E-05	4.05E-08
0.06	1.51E+01	1.83E-05	5.61E-05	3.64E-08
0.08	9.44E+02	1.99E-03	7.12E-03	3.14E-06
0.1	1.55E+02	4.65E-04	1.71E-03	7.11E-07
0.15	9.19E+01	4.85E-04	1.62E-03	7.99E-07
0.2	1.19E+03	9.17E-03	2.74E-02	1.62E-05
0.3	5.70E+02	7.48E-03	1.89E-02	1.42E-05
0.4	5.18E+01	9.91E-04	2.22E-03	1.93E-06
0.5	3.03E+02	7.79E-03	1.60E-02	1.53E-05
0.6	6.95E+02	2.27E-02	4.37E-02	4.43E-05
0.8	6.97E+02	3.31E-02	5.78E-02	6.30E-05
1	1.27E+03	8.11E-02	1.32E-01	1.50E-04
1.5	2.93E+02	3.16E-02	4.62E-02	5.32E-05
2	6.64E+00	1.03E-03	1.41E-03	1.59E-06
3	7.84E+02	2.01E-02	2.56E-01	2.72E-04
				3.47E-04

Minimum Detectable Exposure Rate =

MDCR<sub>surveyor</sub>(cpm/μR/hr) = 2028 cpm/μR/hr  
0.919 μR/hr

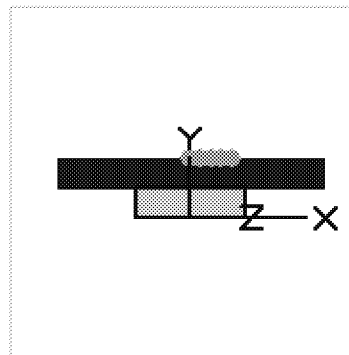
and MDC for Ra-226 based on a normalized 1 pCi/g in soil with density of 1.6 g/cm<sup>3</sup>

Scan MDC = 0.92 pCi/g  
3.30E+01 Bq/kg

RSI  
Ra-226 Calculation

Case Summary of TI1BD

MicroShield 8.02 CB&I Federal Services (8.02-0000)				
Date		By		Checked
Filename		Run Date	Run Time	Duration
TI1BD 20150310.msd		March 10, 2015	2:28:50 PM	00:00:01
Project Info				
Case Title		TI1BD		
Description		RSI Soil Distributed Source, no cover		
Geometry		8 - Cylinder Volume - End Shields		
Source Dimensions				
Height		15.0 cm (5.9 in)		
Radius		28.0 cm (11.0 in)		
Dose Points				
A	X	Y	Z	
#1	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	0.0 cm (0 in)	
#2	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	15.24 cm (6.0 in)	
#3	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	30.48 cm (1 ft)	
#4	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	45.72 cm (1 ft 6.0 in)	
#5	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	60.96 cm (2 ft)	
Shields				
Shield N	Dimension	Material	Density	
Source	3.69e+04 cm³	FGR 12 Soil	1.6	
Shield 1	15.24 cm	Air	0.00122	
Shield 2	.318 cm	Carbon	2.27	
Shield 3	.051 cm	Aluminum	2.7	
Air Gap		Air	0.00122	
Immersion		Air	0.00122	
Source Input: Grouping Method - Standard Indices				
Number of Groups: 25				
Lower Energy Cutoff: 0.015				
Photons < 0.015: Included				
Library: Grove				
Nuclide	Ci	Bq	µCi/cm³	Bq/cm³
Bi-210	5.2037e-008	1.9254e+003	1.4085e-006	5.2115e-002
Bi-214	7.2607e-008	2.6865e+003	1.9653e-006	7.2715e-002
Pb-210	5.2050e-008	1.9258e+003	1.4088e-006	5.2127e-002
Pb-214	7.2607e-008	2.6865e+003	1.9653e-006	7.2715e-002
Po-210	5.1681e-008	1.9122e+003	1.3989e-006	5.1758e-002
Po-214	7.2592e-008	2.6859e+003	1.9648e-006	7.2699e-002
Po-218	7.2621e-008	2.6870e+003	1.9657e-006	7.2729e-002
Ra-226	7.2621e-008	2.6870e+003	1.9656e-006	7.2729e-002



Rn-222	7.2621e-008	2.6870e+003	1.9657e-006	7.2729e-002	
Buildup: The material reference is Source					
Integration Parameters					
Radial				20	
Circumferential				10	
Y Direction (axial)				10	
Results - Dose Point # 1 - (0,30.609,0) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm²/sec No Buildup	Fluence Rate MeV/cm²/sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	8.668e+02	1.018e-06	1.134e-06	8.731e-08	9.731e-08
0.05	1.077e+02	5.454e-05	1.533e-04	1.453e-07	4.085e-07
0.08	6.194e+02	8.500e-04	3.361e-03	1.345e-06	5.319e-06
0.1	3.647e+00	7.163e-06	2.883e-05	1.096e-08	4.411e-08
0.2	2.894e+02	1.493e-03	4.764e-03	2.636e-06	8.408e-06
0.3	5.544e+02	4.920e-03	1.306e-02	9.333e-06	2.478e-05
0.4	1.028e+03	1.341e-02	3.139e-02	2.613e-05	6.117e-05
0.5	4.799e+01	8.438e-04	1.799e-03	1.656e-06	3.532e-06
0.6	1.295e+03	2.905e-02	5.764e-02	5.671e-05	1.125e-04
0.8	2.539e+02	8.353e-03	1.490e-02	1.589e-05	2.835e-05
1.0	8.411e+02	3.720e-02	6.180e-02	6.856e-05	1.139e-04
1.5	5.115e+02	3.847e-02	5.690e-02	6.473e-05	9.573e-05
2.0	7.189e+02	7.814e-02	1.083e-01	1.208e-04	1.674e-04
Totals	7.138e+03	2.128e-01	3.541e-01	3.681e-04	6.217e-04
Results - Dose Point # 2 - (0,30.609,15.24) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm²/sec No Buildup	Fluence Rate MeV/cm²/sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	8.668e+02	5.024e-07	5.694e-07	4.309e-08	4.884e-08
0.05	1.077e+02	4.681e-05	1.312e-04	1.247e-07	3.496e-07
0.08	6.194e+02	7.279e-04	2.878e-03	1.152e-06	4.555e-06
0.1	3.647e+00	6.133e-06	2.472e-05	9.382e-09	3.783e-08
0.2	2.894e+02	1.279e-03	4.107e-03	2.257e-06	7.249e-06
0.3	5.544e+02	4.216e-03	1.129e-02	7.998e-06	2.142e-05
0.4	1.028e+03	1.150e-02	2.718e-02	2.241e-05	5.296e-05
0.5	4.799e+01	7.242e-04	1.559e-03	1.422e-06	3.061e-06
0.6	1.295e+03	2.495e-02	4.999e-02	4.871e-05	9.758e-05
0.8	2.539e+02	7.184e-03	1.294e-02	1.366e-05	2.461e-05
1.0	8.411e+02	3.203e-02	5.372e-02	5.904e-05	9.903e-05
1.5	5.115e+02	3.321e-02	4.954e-02	5.588e-05	8.336e-05
2.0	7.189e+02	6.758e-02	9.439e-02	1.045e-04	1.460e-04
Totals	7.138e+03	1.835e-01	3.078e-01	3.172e-04	5.402e-04
Results - Dose Point # 3 - (0,30.609,30.48) cm					

Energy (MeV)	Activity (Photons/sec)	Fluence Rate	Fluence Rate	Exposure Rate	Exposure Rate
		MeV/cm <sup>2</sup> /sec No Buildup	MeV/cm <sup>2</sup> /sec With Buildup	mR/hr No Buildup	mR/hr With Buildup
0.015	8.668e+02	1.807e-07	2.053e-07	1.550e-08	1.761e-08
0.05	1.077e+02	2.461e-05	7.133e-05	6.557e-08	1.900e-07
0.08	6.194e+02	3.925e-04	1.646e-03	6.212e-07	2.605e-06
0.1	3.647e+00	3.335e-06	1.442e-05	5.102e-09	2.207e-08
0.2	2.894e+02	7.102e-04	2.483e-03	1.254e-06	4.382e-06
0.3	5.544e+02	2.371e-03	6.904e-03	4.498e-06	1.310e-05
0.4	1.028e+03	6.532e-03	1.673e-02	1.273e-05	3.260e-05
0.5	4.799e+01	4.147e-04	9.643e-04	8.139e-07	1.893e-06
0.6	1.295e+03	1.439e-02	3.102e-02	2.808e-05	6.055e-05
0.8	2.539e+02	4.190e-03	8.076e-03	7.970e-06	1.536e-05
1.0	8.411e+02	1.886e-02	3.366e-02	3.476e-05	6.205e-05
1.5	5.115e+02	1.990e-02	3.130e-02	3.348e-05	5.266e-05
2.0	7.189e+02	4.098e-02	5.999e-02	6.338e-05	9.277e-05
<b>Totals</b>	<b>7.138e+03</b>	<b>1.088e-01</b>	<b>1.929e-01</b>	<b>1.877e-04</b>	<b>3.382e-04</b>

**Results - Dose Point # 4 - (0,30.609,45.72) cm**

Energy (MeV)	Activity (Photons/sec)	Fluence Rate	Fluence Rate	Exposure Rate	Exposure Rate
		MeV/cm <sup>2</sup> /sec No Buildup	MeV/cm <sup>2</sup> /sec With Buildup	mR/hr No Buildup	mR/hr With Buildup
0.015	8.668e+02	6.903e-08	7.825e-08	5.921e-09	6.712e-09
0.05	1.077e+02	1.169e-05	3.476e-05	3.114e-08	9.261e-08
0.08	6.194e+02	1.866e-04	8.010e-04	2.952e-07	1.268e-06
0.1	3.647e+00	1.586e-06	7.051e-06	2.427e-09	1.079e-08
0.2	2.894e+02	3.401e-04	1.241e-03	6.002e-07	2.190e-06
0.3	5.544e+02	1.142e-03	3.491e-03	2.167e-06	6.621e-06
0.4	1.028e+03	3.163e-03	8.525e-03	6.163e-06	1.661e-05
0.5	4.799e+01	2.018e-04	4.942e-04	3.961e-07	9.701e-07
0.6	1.295e+03	7.032e-03	1.598e-02	1.373e-05	3.119e-05
0.8	2.539e+02	2.065e-03	4.193e-03	3.927e-06	7.975e-06
1.0	8.411e+02	9.359e-03	1.758e-02	1.725e-05	3.241e-05
1.5	5.115e+02	1.002e-02	1.653e-02	1.686e-05	2.781e-05
2.0	7.189e+02	2.087e-02	3.194e-02	3.228e-05	4.939e-05
<b>Totals</b>	<b>7.138e+03</b>	<b>5.440e-02</b>	<b>1.008e-01</b>	<b>9.371e-05</b>	<b>1.765e-04</b>

**Results - Dose Point # 5 - (0,30.609,60.96) cm**

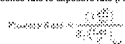
Energy (MeV)	Activity (Photons/sec)	Fluence Rate	Fluence Rate	Exposure Rate	Exposure Rate
		MeV/cm <sup>2</sup> /sec No Buildup	MeV/cm <sup>2</sup> /sec With Buildup	mR/hr No Buildup	mR/hr With Buildup
0.015	8.668e+02	2.567e-08	2.916e-08	2.202e-09	2.501e-09
0.05	1.077e+02	5.697e-06	1.777e-05	1.518e-08	4.733e-08
0.08	6.194e+02	9.313e-05	4.254e-04	1.474e-07	6.732e-07
0.1	3.647e+00	7.972e-07	3.776e-06	1.220e-09	5.777e-09
0.2	2.894e+02	1.739e-04	6.730e-04	3.070e-07	1.188e-06



Case Summary of TIIBD

0.3	5.544e+02	5.901e-04	1.903e-03	1.119e-06	3.610e-06
0.4	1.028e+03	1.646e-03	4.666e-03	3.208e-06	9.092e-06
0.5	4.799e+01	1.056e-04	2.714e-04	2.073e-07	5.327e-07
0.6	1.295e+03	3.699e-03	8.800e-03	7.220e-06	1.718e-05
0.8	2.539e+02	1.095e-03	2.320e-03	2.082e-06	4.412e-06
1.0	8.411e+02	4.994e-03	9.766e-03	9.205e-06	1.800e-05
1.5	5.115e+02	5.414e-03	9.260e-03	9.109e-06	1.558e-05
2.0	7.189e+02	1.138e-02	1.800e-02	1.760e-05	2.784e-05
<b>Totals</b>	<b>7.138e+03</b>	<b>2.920e-02</b>	<b>5.611e-02</b>	<b>5.022e-05</b>	<b>9.816e-05</b>

RSI NaI Scan for Ra-226 @ 1pCi/g, 0 inches soil, 15 cm thick x 28 cm Radius, 0" offset  
Fluence rate to exposure rate (FRER, no units) =  $(1 \text{ uR/hr}) / (E \cdot \mu_{en} / \rho)_{air}$



Mass energy-absorption coefficients from 1970 Rad Health Handbook

TABLE 1		
Energy, keV	$(\mu_{en}/\rho)_{air}$ , cm <sup>2</sup> /g	FRER
15	1.35	0.0517
20	0.518	0.0969
30	0.147	0.2268
40	0.084	0.3906
50	0.0584	0.5208
60	0.0292	0.5708
80	0.0238	0.5297
100	0.0231	0.4329
150	0.0251	0.2656
200	0.0268	0.1668
300	0.0289	0.1157
400	0.0296	0.0845
500	0.0297	0.0573
600	0.0296	0.0563
800	0.0289	0.0433
1,000	0.0280	0.0357
1,500	0.0295	0.0261
2,000	0.0234	0.0214

Probability of interaction (P) through end of detector for given energy is

$$P = 1 - e^{-\mu_{en} \cdot x}$$

For RSI-1: 10.16 cm X 10.16 cm X 40.64 cm

carbon fiber case 0.318 cm thick; Al wrap 0.051 cm

$$\mu_{en} = 3.67 \text{ g/cm}^3$$

Mass attenuation coefficients from 1970 Rad Health Handbook

TABLE 2		
Energy, keV	$(\mu/\rho)_{air}$ , cm <sup>2</sup> /g	P
15	47.4	1.00
20	22.3	1.00
30	7.45	1.00
40	19.3	1.00
50	10.7	1.00
60	6.62	1.00
80	3.12	1.00
100	1.72	1.00
150	0.625	1.00
200	0.334	1.00
300	0.187	1.00
400	0.117	0.99
500	0.0965	0.97
600	0.0828	0.95
800	0.0616	0.92
1,000	0.0586	0.89
1,500	0.0469	0.83
2,000	0.0415	0.79

Relative Detector Response (RDR) = relative fluence-to-exposure rate (FRER) times probability (P) of interaction

Relative detector response (RDR) is the relative detector response to the source

TABLE 3			
Energy, keV	Fluence Rate	P	RDR
15	0.0517	1.00	0.0517
20	0.0969	1.00	0.0969
30	0.2268	1.00	0.2268
40	0.3906	1.00	0.3906
50	0.5208	1.00	0.5208
60	0.5708	1.00	0.5708
80	0.5297	1.00	0.5297
100	0.4329	1.00	0.4329
150	0.2656	1.00	0.2656
200	0.1668	1.00	0.1668
300	0.1157	1.00	0.1155
400	0.0845	0.99	0.0834
500	0.0673	0.97	0.0654
600	0.0563	0.95	0.0537
800	0.0433	0.92	0.0398
1,000	0.0357	0.89	0.0317
1,500	0.0261	0.83	0.0216
2,000	0.0214	0.79	0.0168

Estimated RSI-700 4-Liter NaI total gamma response @ 602keV

42483

cpm/uR/hr

Use same methodology and interpolating for Cs-137 response have:

Energy, keV	$(\mu_{en}/\rho)_{air}$ , cm <sup>2</sup> /g	FRER =	0.0514
562	0.0294		
Energy, keV	$(\mu/\rho)_{air}$ , cm <sup>2</sup> /g	Probability =	0.95
562	0.0780		
		RDR =	0.0486

For this detector the response to another energy is based on the ratio of the relative detector response, RDR, to the Cs-137 energy

$$RDR_{Cs-137} = \frac{RDR_{Cs-137}}{RDR_{Cs-137}} = 1$$

TABLE 4		
Energy, keV	RDR <sub>air</sub>	RSI NaI cpm per $\mu\text{R/hr}$ , $E_s$
15	0.0517	43202
20	0.0969	84753
30	0.2268	198333
40	0.3906	341660
50	0.5208	435547
60	0.5708	499220
80	0.5297	461268
100	0.4329	378877
150	0.2656	222311
200	0.1668	160180
300	0.1155	101073
400	0.0834	72911
500	0.0654	57226
600	0.0537	46985
800	0.0486	42483
1,000	0.0398	34789
1,500	0.0317	27724
2,000	0.0216	18688
	0.0168	14682

RSI NaI Scan for Ra-226 @ 1pCi/g, 0 inches soil, 15 cm thick x 28 cm Radius, 0" offset

Assumptions

Detector is 4 in. (10.16 cm) X 4 in. X 16 in.

15.24 cm detector height

15 x 28 source at 1.6 g/cm<sup>3</sup> (dry soil) - Buildup reference

No soil cover

0.318 carbon casing @ 2.27 g/cm<sup>3</sup>

0.051 cm aluminum wrap @ 2.7 g/cm<sup>3</sup>

Average background count rate observed to be 3,787 cpi (225,018 cpm) in 5.3 uR/hr field (RDR 10, Gross Counts)

42,483 cpm/uR/hr

Centerpoint of detector has no offset from center of source

MDCR<sub>survey</sub> for Cs-137 energy  
42463 cpm/μR/hr  
Bgnd = 226016 cpm  
minimum detectable exposure rate, Cs-137 = 0.21 μR/hr

Observation Interval, i = 2 second  
b<sub>1</sub> = 7533.9 counts  
d' = 3.28 from Table 5.1 of NUREG-1507  
MDCR = 8940.9 cpm  
MDCR<sub>survey</sub> = 9003 cpm

Table 5				
Energy, keV	MicroShield Exposure Rate, μR/hr (with buildup)	cpm/μR/hr	cpm/μR/hr (without buildup)	Percent of NaI detector response
15	9.79E-06	45302	7	0.0%
20	0.000E+00	84753	0	0.0%
30	0.000E+00	198333	0	0.0%
40	0.000E+00	341660	0	0.0%
50	4.069E-04	455547	299	0.7%
60	0.000E+00	499250	0	0.0%
80	5.319E-03	463265	3954	9.9%
100	4.411E-05	376637	27	0.1%
150	0.000E+00	232311	0	0.0%
200	6.406E-03	163160	2207	5.5%
300	5.479E-02	101033	4027	10.1%
400	6.117E-02	72931	7176	17.9%
500	3.532E-03	67226	325	0.8%
600	1.129E-01	46965	9503	21.2%
800	2.839E-02	34769	1567	4.0%
1000	1.139E-01	27724	5080	12.7%
1500	9.573E-02	19866	2909	7.3%
2000	1.674E-01	14662	3954	9.9%
Total	6.216E-01		40065	100.0%

$$S_i = d' \sqrt{b_i}$$

$$MDCR = S_i (60 / i)$$

$$MDCR_{SURVEY} = \frac{MDCR}{\sqrt{P}}$$

P=0.90

mR/hr, from MS	
15	9.79E-06
20	
30	
40	
50	4.09E-07
60	
80	5.32E-05
100	4.41E-08
150	
200	8.41E-05
300	2.48E-05
400	6.12E-05
500	3.59E-05
600	1.13E-04
800	2.94E-05
1000	1.14E-04
1500	9.57E-05
2000	1.67E-04
	6.216E-04

MicroShield Output					
0.015	8.97E+02	1.02E-05	1.13E-06	6.73E-06	9.73E-08
0.25	1.09E+02	1.43E-05	1.33E-04	1.43E-07	4.09E-07
0.5	6.13E+02	6.53E-04	3.35E-03	1.55E-06	5.24E-06
0.1	3.65E+00	7.15E-06	2.88E-05	1.10E-08	4.41E-08
0.2	2.89E+02	1.49E-03	4.76E-03	2.64E-05	8.41E-05
0.3	5.54E+02	4.92E-03	1.31E-02	9.33E-06	2.48E-05
0.4	1.03E+03	1.94E-02	3.14E-02	2.61E-05	6.12E-05
0.5	4.80E+01	5.44E-04	1.63E-03	1.66E-05	3.59E-05
0.6	1.30E+03	2.91E-02	5.76E-02	5.67E-05	1.13E-04
0.8	2.54E+02	8.73E-03	1.49E-02	1.89E-05	2.64E-05
1	6.41E+02	3.72E-02	6.18E-02	6.86E-05	1.14E-04
1.5	3.12E+02	3.65E-02	5.69E-02	6.47E-05	9.57E-05
2	7.19E+02	7.81E-02	1.08E-01	1.21E-04	1.67E-04

Minimum Detectable Exposure Rate =

MDCR<sub>survey</sub>(cpm/μR/hr) = 9003 cpm/μR/hr  
0.225 μR/hr

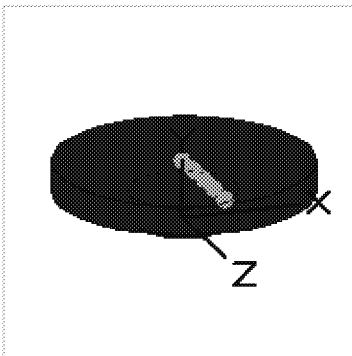
and MDC for Ra-226 based on a normalized 1 pCi/g in soil with density of 1.6

Scan MDC = 0.336 pCi/g

1.34E+01 Bq/kg

RSI  
Cs-137 Calculation

MicroShield 8.02 CB&I Federal Services (8.02-0000)			
<b>Date</b>	<b>By</b>	<b>Checked</b>	
<b>Filename</b>	<b>Run Date</b>	<b>Run Time</b>	<b>Duration</b>
TI1BD 20150310.msd	September 25, 2018	1:39:34 PM	00:00:00
<b>Project Info</b>			
Case Title	TI1BD Cs-137		
Description	RSI Soil Distributed Source, no cover		
Geometry	8 - Cylinder Volume - End Shields		
<b>Source Dimensions</b>			
Height	15.0 cm (5.9 in)		
Radius	28.0 cm (11.0 in)		
<b>Dose Points</b>			
A	X	Y	Z
#1	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	0.0 cm (0 in)
#2	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	15.24 cm (6.0 in)
#3	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	30.48 cm (1 ft)
#4	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	45.72 cm (1 ft 6.0 in)
#5	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	60.96 cm (2 ft)
<b>Shields</b>			
Shield N	Dimension	Material	Density
Source	3.69e+04 cm <sup>3</sup>	FRG12Soil (mass fxn)	1.6
Shield 1	15.24 cm	Air	0.00122
Shield 2	.318 cm	Carbon	2.27
Shield 3	.051 cm	Aluminum	2.7
Air Gap		Air	0.00122
Immersion		Air	0.00122
<b>Source Input: Grouping Method - Standard Indices</b>			
Number of Groups: 25			
Lower Energy Cutoff: 0.015			
Photons < 0.015: Included			
Library: Grove			
Nuclide	Ci	Bq	μCi/cm <sup>3</sup>
Ba-137m	5.5920e-008	2.0690e+003	1.5136e-006
Cs-137	5.9112e-008	2.1872e+003	1.6000e-006
<b>Buildup: The material reference is Source</b>			
<b>Integration Parameters</b>			
Radial			20
Circumferential			10
Y Direction (axial)			10



Results - Dose Point # 1 - (0,30.609,0) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	2.148e+01	2.522e-08	2.811e-08	2.164e-09	2.411e-09
0.03	1.219e+02	1.098e-05	1.714e-05	1.088e-07	1.699e-07
0.04	2.876e+01	7.548e-06	1.572e-05	3.338e-08	6.953e-08
0.6	1.862e+03	4.176e-02	8.285e-02	8.151e-05	1.617e-04
<b>Totals</b>	<b>2.034e+03</b>	<b>4.178e-02</b>	<b>8.288e-02</b>	<b>8.165e-05</b>	<b>1.620e-04</b>

Results - Dose Point # 2 - (0,30.609,15.24) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	2.148e+01	1.245e-08	1.411e-08	1.068e-09	1.210e-09
0.03	1.219e+02	9.454e-06	1.477e-05	9.369e-08	1.464e-07
0.04	2.876e+01	6.491e-06	1.351e-05	2.871e-08	5.976e-08
0.6	1.862e+03	3.587e-02	7.186e-02	7.001e-05	1.403e-04
<b>Totals</b>	<b>2.034e+03</b>	<b>3.588e-02</b>	<b>7.188e-02</b>	<b>7.013e-05</b>	<b>1.405e-04</b>

Results - Dose Point # 3 - (0,30.609,30.48) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	2.148e+01	4.478e-09	5.088e-09	3.841e-10	4.364e-10
0.03	1.219e+02	4.698e-06	7.471e-06	4.656e-08	7.404e-08
0.04	2.876e+01	3.354e-06	7.160e-06	1.483e-08	3.167e-08
0.6	1.862e+03	2.068e-02	4.459e-02	4.036e-05	8.704e-05
<b>Totals</b>	<b>2.034e+03</b>	<b>2.069e-02</b>	<b>4.460e-02</b>	<b>4.043e-05</b>	<b>8.714e-05</b>

Results - Dose Point # 4 - (0,30.609,45.72) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	2.148e+01	1.710e-09	1.939e-09	1.467e-10	1.663e-10
0.03	1.219e+02	2.159e-06	3.491e-06	2.140e-08	3.460e-08
0.04	2.876e+01	1.582e-06	3.460e-06	6.999e-09	1.530e-08
0.6	1.862e+03	1.011e-02	2.297e-02	1.973e-05	4.483e-05
<b>Totals</b>	<b>2.034e+03</b>	<b>1.011e-02</b>	<b>2.298e-02</b>	<b>1.976e-05</b>	<b>4.488e-05</b>

Results - Dose Point # 5 - (0,30.609,60.96) cm					
Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	2.148e+01	6.360e-10	7.226e-10	5.455e-11	6.198e-11
0.03	1.219e+02	9.701e-07	1.613e-06	9.615e-09	1.598e-08
0.04	2.876e+01	7.545e-07	1.721e-06	3.337e-09	7.610e-09
0.6	1.862e+03	5.317e-03	1.265e-02	1.038e-05	2.469e-05

<b>Totals</b>	<b>2.034e+03</b>	<b>5.318e-03</b>	<b>1.265e-02</b>	<b>1.039e-05</b>	<b>2.471e-05</b>
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Centerpoint of detector has no offset from center of source

Energy, keV	RDC <sub>c</sub>	RSI Nat cpm per keV/hr, E <sub>c</sub>
15	0.0617	452.02
20	0.0969	547.53
30	0.2758	1028.03
40	0.3906	1415.60
50	0.5206	855.57
60	0.5740	4992.30
80	0.5297	463.28
100	0.4328	212.87
150	0.2586	212.11
200	0.1866	163.80
300	0.1155	104.03
400	0.0634	72.91
500	0.0654	572.26
600	0.0937	469.05
800	0.0466	422.03
900	0.0396	347.89
1,000	0.0317	277.24
1,500	0.0216	189.88
2,000	0.0163	146.63



MDCR<sub>survey</sub> for Cs-137 energy  
42453 cpm/μR/hr  
Bgkd = 225018 cpm  
Observation Interval,  $t_i = 2$  second  
 $b_i = 7533.9$  counts  
 $\sigma_i^2 = 3.28$  from Table 6.1 of NUREG-1507  
MDCR = 8540.9 cpm  
MDCR<sub>survey</sub> = 9003 cpm  
minimum detectable exposure rate, Cs-137 = 0.21 μR/hr

Table 5				
Energy, keV	Microshield Exposure Rate, μR/hr (with buildup)	cpm/μR/hr	cpm/μR/hr (weighted)	Percent of total detected exposure
15	2.41E-05	45202	1	0.0%
20	0.000E+00	64753	0	0.0%
30	1.099E-04	198333	208	0.4%
40	8.953E-05	341660	147	0.3%
50	0.000E+00	455647	0	0.0%
60	0.000E+00	499230	0	0.0%
80	0.000E+00	463289	0	0.0%
100	0.000E+00	378637	0	0.0%
150	0.000E+00	232311	0	0.0%
200	0.000E+00	183180	0	0.0%
300	0.000E+00	101033	0	0.0%
400	0.000E+00	72931	0	0.0%
500	0.000E+00	57226	0	0.0%
600	1.517E-01	46985	46915	99.2%
800	0.000E+00	34759	0	0.0%
1000	0.000E+00	27724	0	0.0%
1500	0.000E+00	18888	0	0.0%
2000	0.000E+00	14652	0	0.0%
Total	1.519E-01		47270	100.0%

$$S_i = d^{-1} \sqrt{b_i}$$

$$MDCR = S_i (60 / i)$$

$$MDCR_{SURVEYOR} = \frac{MDCR}{\sqrt{P}}$$

P=0.90

mR/hr, from MS	
15	2.41E-09
20	
30	1.698E-07
40	6.953E-05
50	
60	
80	
100	
150	
200	
300	
400	
500	
600	1.62E-04
800	
1000	
1500	
2000	
1.519E-04	

Microshield Output					
0.015	2.15E+01	2.52E-08	2.81E-08	2.16E-09	2.41E-09
0.03	1.22E+02	1.10E-05	1.71E-05	1.09E-07	1.70E-07
0.04	2.88E+01	7.55E-06	1.57E-05	3.34E-08	6.36E-08
0.1					
0.2					
0.3					
0.4					
0.5					
0.6	1.86E+03	4.18E-02	8.29E-02	8.15E-05	1.62E-04
1					
1.5					
2					

Minimum Detectable Exposure Rate =

MDCR surveyor(cpm/μR/hr) = 9003  
0.190 μR/hr

and MDC for Ra-226 based on a normalized 1 pCi/g in soil with density of 1.6

Count Rate (cpm) = 4.35E+01

Scan MDC = 1.18 pCi/g  
4.35E+01 Bq/kg

RSI  
Th-232 Calculation

**MicroShield 8.02  
Aptim (8.02-0000)**

Date	By	Checked

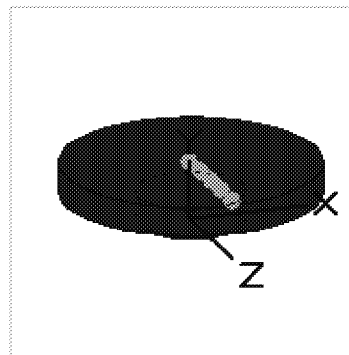
Filename	Run Date	Run Time	Duration
T11BD 20150310.msd	September 4, 2019	8:25:25 AM	00:00:01

Project Info	
Case Title	T11BD
Description	RSI Soil Distributed Source, no cover
Geometry	8 - Cylinder Volume - End Shields

Source Dimensions	
Height	15.0 cm (5.9 in)
Radius	28.0 cm (11.0 in)

Dose Points			
A	X	Y	Z
#1	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	0.0 cm (0 in)
#2	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	15.24 cm (6.0 in)
#3	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	30.48 cm (1 ft)
#4	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	45.72 cm (1 ft 6.0 in)
#5	0.0 cm (0 in)	30.609 cm (1 ft 0.1 in)	60.96 cm (2 ft)

Shields			
Shield N	Dimension	Material	Density
Source	3.69e+04 cm <sup>3</sup>	FGR12 Soil	1.6
Shield 1	15.24 cm	Air	0.00122
Shield 2	.318 cm	Carbon	2.27
Shield 3	.051 cm	Aluminum	2.7
Air Gap		Air	0.00122
Immersion		Air	0.00122



Source Input: Grouping Method - Standard Indices				
Number of Groups: 25				
Lower Energy Cutoff: 0.015				
Photons < 0.015: Included				
Library: Grove				
Nuclide	Ci	Bq	μCi/cm <sup>3</sup>	Bq/cm <sup>3</sup>
Ac-228	5.9112e-008	2.1871e+003	1.6000e-006	5.9200e-002
Bi-212	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Pb-212	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Po-212	3.7873e-008	1.4013e+003	1.0251e-006	3.7929e-002
Po-216	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Ra-224	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Ra-228	5.9112e-008	2.1871e+003	1.6000e-006	5.9200e-002
Rn-220	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002

Th-228	5.9112e-008	2.1871e+003	1.6000e-006	5.9199e-002
Th-232	5.9112e-008	2.1872e+003	1.6000e-006	5.9200e-002
Tl-208	2.1239e-008	7.8584e+002	5.7487e-007	2.1270e-002

**Buildup: The material reference is Source  
Integration Parameters**

Radial	20
Circumferential	10
Y Direction (axial)	10

**Results - Dose Point # 1 - (0,30.609,0) cm**

Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	1.787e+03	2.099e-06	2.339e-06	1.800e-07	2.006e-07
0.04	2.236e+01	5.870e-06	1.222e-05	2.596e-08	5.407e-08
0.06	1.512e+01	1.188e-05	4.010e-05	2.360e-08	7.964e-08
0.08	9.439e+02	1.295e-03	5.122e-03	2.050e-06	8.105e-06
0.1	1.552e+02	3.049e-04	1.227e-03	4.664e-07	1.877e-06
0.15	9.193e+01	3.215e-04	1.164e-03	5.295e-07	1.917e-06
0.2	1.188e+03	6.131e-03	1.956e-02	1.082e-05	3.452e-05
0.3	5.702e+02	5.060e-03	1.344e-02	9.599e-06	2.549e-05
0.4	5.175e+01	6.750e-04	1.580e-03	1.315e-06	3.079e-06
0.5	3.034e+02	5.334e-03	1.137e-02	1.047e-05	2.233e-05
0.6	6.954e+02	1.560e-02	3.095e-02	3.045e-05	6.041e-05
0.8	6.969e+02	2.293e-02	4.091e-02	4.361e-05	7.781e-05
1.0	1.274e+03	5.632e-02	9.358e-02	1.038e-04	1.725e-04
1.5	2.934e+02	2.207e-02	3.263e-02	3.713e-05	5.491e-05
2.0	6.638e+00	7.214e-04	9.995e-04	1.116e-06	1.546e-06
3.0	7.843e+02	1.410e-01	1.810e-01	1.913e-04	2.456e-04
<b>Totals</b>	<b>8.879e+03</b>	<b>2.778e-01</b>	<b>4.336e-01</b>	<b>4.429e-04</b>	<b>7.104e-04</b>

**Results - Dose Point # 2 - (0,30.609,15.24) cm**

Energy (MeV)	Activity (Photons/sec)	Fluence Rate MeV/cm <sup>2</sup> /sec No Buildup	Fluence Rate MeV/cm <sup>2</sup> /sec With Buildup	Exposure Rate mR/hr No Buildup	Exposure Rate mR/hr With Buildup
0.015	1.787e+03	1.036e-06	1.174e-06	8.884e-08	1.007e-07
0.04	2.236e+01	5.048e-06	1.051e-05	2.232e-08	4.647e-08
0.06	1.512e+01	1.019e-05	3.428e-05	2.023e-08	6.809e-08
0.08	9.439e+02	1.109e-03	4.386e-03	1.755e-06	6.941e-06
0.1	1.552e+02	2.610e-04	1.052e-03	3.993e-07	1.610e-06
0.15	9.193e+01	2.753e-04	1.001e-03	4.533e-07	1.649e-06
0.2	1.188e+03	5.251e-03	1.686e-02	9.267e-06	2.976e-05
0.3	5.702e+02	4.337e-03	1.161e-02	8.227e-06	2.203e-05
0.4	5.175e+01	5.789e-04	1.368e-03	1.128e-06	2.666e-06
0.5	3.034e+02	4.578e-03	9.857e-03	8.986e-06	1.935e-05
0.6	6.954e+02	1.340e-02	2.684e-02	2.615e-05	5.239e-05

0.8	6.969e+02	1.972e-02	3.552e-02	3.751e-05	6.757e-05
1.0	1.274e+03	4.850e-02	8.134e-02	8.940e-05	1.499e-04
1.5	2.934e+02	1.905e-02	2.842e-02	3.205e-05	4.781e-05
2.0	6.638e+00	6.239e-04	8.715e-04	9.649e-07	1.348e-06
3.0	7.843e+02	1.223e-01	1.580e-01	1.659e-04	2.144e-04
<b>Totals</b>	<b>8.879e+03</b>	<b>2.400e-01</b>	<b>3.772e-01</b>	<b>3.823e-04</b>	<b>6.177e-04</b>

**Results - Dose Point # 3 - (0,30.609,30.48) cm**

Energy (MeV)	Activity (Photons/sec)	Fluence Rate	Fluence Rate	Exposure Rate	Exposure Rate
		MeV/cm <sup>2</sup> /sec No Buildup	MeV/cm <sup>2</sup> /sec With Buildup	mR/hr No Buildup	mR/hr With Buildup
0.015	1.787e+03	3.726e-07	4.233e-07	3.196e-08	3.631e-08
0.04	2.236e+01	2.608e-06	5.567e-06	1.153e-08	2.462e-08
0.06	1.512e+01	5.418e-06	1.903e-05	1.076e-08	3.779e-08
0.08	9.439e+02	5.982e-04	2.509e-03	9.467e-07	3.970e-06
0.1	1.552e+02	1.419e-04	6.138e-04	2.171e-07	9.391e-07
0.15	9.193e+01	1.516e-04	5.985e-04	2.496e-07	9.855e-07
0.2	1.188e+03	2.916e-03	1.019e-02	5.146e-06	1.799e-05
0.3	5.702e+02	2.439e-03	7.101e-03	4.626e-06	1.347e-05
0.4	5.175e+01	3.288e-04	8.422e-04	6.406e-07	1.641e-06
0.5	3.034e+02	2.621e-03	6.096e-03	5.145e-06	1.196e-05
0.6	6.954e+02	7.725e-03	1.666e-02	1.508e-05	3.251e-05
0.8	6.969e+02	1.150e-02	2.217e-02	2.188e-05	4.217e-05
1.0	1.274e+03	2.855e-02	5.097e-02	5.263e-05	9.395e-05
1.5	2.934e+02	1.141e-02	1.795e-02	1.920e-05	3.021e-05
2.0	6.638e+00	3.784e-04	5.539e-04	5.851e-07	8.565e-07
3.0	7.843e+02	7.532e-02	1.012e-01	1.022e-04	1.373e-04
<b>Totals</b>	<b>8.879e+03</b>	<b>1.441e-01</b>	<b>2.375e-01</b>	<b>2.286e-04</b>	<b>3.880e-04</b>

**Results - Dose Point # 4 - (0,30.609,45.72) cm**

Energy (MeV)	Activity (Photons/sec)	Fluence Rate	Fluence Rate	Exposure Rate	Exposure Rate
		MeV/cm <sup>2</sup> /sec No Buildup	MeV/cm <sup>2</sup> /sec With Buildup	mR/hr No Buildup	mR/hr With Buildup
0.015	1.787e+03	1.423e-07	1.613e-07	1.221e-08	1.384e-08
0.04	2.236e+01	1.230e-06	2.691e-06	5.442e-09	1.190e-08
0.06	1.512e+01	2.575e-06	9.266e-06	5.114e-09	1.840e-08
0.08	9.439e+02	2.843e-04	1.221e-03	4.499e-07	1.932e-06
0.1	1.552e+02	6.751e-05	3.001e-04	1.033e-07	4.591e-07
0.15	9.193e+01	7.233e-05	2.964e-04	1.191e-07	4.881e-07
0.2	1.188e+03	1.396e-03	5.094e-03	2.464e-06	8.991e-06
0.3	5.702e+02	1.175e-03	3.590e-03	2.228e-06	6.810e-06
0.4	5.175e+01	1.592e-04	4.291e-04	3.102e-07	8.362e-07
0.5	3.034e+02	1.275e-03	3.124e-03	2.504e-06	6.133e-06
0.6	6.954e+02	3.776e-03	8.579e-03	7.370e-06	1.675e-05
0.8	6.969e+02	5.667e-03	1.151e-02	1.078e-05	2.189e-05
1.0	1.274e+03	1.417e-02	2.662e-02	2.612e-05	4.907e-05
1.5	2.934e+02	5.748e-03	9.482e-03	9.672e-06	1.595e-05

2.0	6.638e+00	1.927e-04	2.949e-04	2.980e-07	4.560e-07
3.0	7.843e+02	3.897e-02	5.443e-02	5.287e-05	7.385e-05
<b>Totals</b>	<b>8.879e+03</b>	<b>7.295e-02</b>	<b>1.250e-01</b>	<b>1.153e-04</b>	<b>2.037e-04</b>

**Results - Dose Point # 5 - (0,30.609,60.96) cm**

Energy (MeV)	Activity (Photons/sec)	Fluence Rate	Fluence Rate	Exposure Rate	Exposure Rate
		MeV/cm <sup>2</sup> /sec No Buildup	MeV/cm <sup>2</sup> /sec With Buildup	mR/hr No Buildup	mR/hr With Buildup
0.015	1.787e+03	5.292e-08	6.013e-08	4.539e-09	5.157e-09
0.04	2.236e+01	5.867e-07	1.338e-06	2.595e-09	5.917e-09
0.06	1.512e+01	1.270e-06	4.826e-06	2.522e-09	9.586e-09
0.08	9.439e+02	1.419e-04	6.483e-04	2.246e-07	1.026e-06
0.1	1.552e+02	3.393e-05	1.607e-04	5.191e-08	2.459e-07
0.15	9.193e+01	3.673e-05	1.601e-04	6.049e-08	2.636e-07
0.2	1.188e+03	7.141e-04	2.763e-03	1.260e-06	4.877e-06
0.3	5.702e+02	6.070e-04	1.957e-03	1.151e-06	3.713e-06
0.4	5.175e+01	8.287e-05	2.349e-04	1.615e-07	4.577e-07
0.5	3.034e+02	6.677e-04	1.716e-03	1.311e-06	3.368e-06
0.6	6.954e+02	1.986e-03	4.724e-03	3.876e-06	9.222e-06
0.8	6.969e+02	3.005e-03	6.368e-03	5.715e-06	1.211e-05
1.0	1.274e+03	7.561e-03	1.479e-02	1.394e-05	2.726e-05
1.5	2.934e+02	3.105e-03	5.311e-03	5.225e-06	8.936e-06
2.0	6.638e+00	1.050e-04	1.662e-04	1.624e-07	2.571e-07
3.0	7.843e+02	2.151e-02	3.094e-02	2.918e-05	4.197e-05
<b>Totals</b>	<b>8.879e+03</b>	<b>3.956e-02</b>	<b>6.994e-02</b>	<b>6.233e-05</b>	<b>1.137e-04</b>

RSI NaI Scan for Th-232 @ 1pCi/g, 0 inches soil, 15 cm thick x 28 cm Radius, 0" offset  
Fluence rate to exposure rate (FRER, no units) =  $\sim (1 \text{ uR/hr})(E\gamma)(u_{\text{air}}/\rho)_{\text{air}}$

$$\text{Fluence Rate} = \frac{A_{\text{source}}}{4\pi r^2} \times \frac{E_{\gamma}}{4\pi r^2} \times \frac{u_{\text{air}}}{\rho_{\text{air}}}$$

Mass energy-absorption coefficients from 1970 Rad Health Handbook

TABLE 1		
Energy, keV	$(u_{\text{air}}/\rho)_{\text{air}}$ , cm <sup>2</sup> /g	FRER
15	1.29	0.0517
20	0.516	0.0969
30	0.147	0.2288
40	0.084	0.3908
50	0.0394	0.5208
60	0.0292	0.5708
80	0.0236	0.5297
100	0.0231	0.4329
150	0.0251	0.2658
200	0.0288	0.1868
300	0.0288	0.1157
400	0.0296	0.0845
500	0.0297	0.0673
600	0.0296	0.0563
800	0.0289	0.0433
1,000	0.0280	0.0357
1,500	0.0255	0.0261
2,000	0.0234	0.0214
3,000	0.0205	0.0163

Probability of Interaction (P) through end of detector for given energy is

$$P = \frac{\mu_{\text{enr}}}{\mu_{\text{enr}} + \mu_{\text{a}}} \times \frac{A_{\text{detector}}}{A_{\text{source}}}$$

For RSI-1: 10.16 cm X 10.16 cm X 40.64 cm carbon fiber case 0.318 cm thick; Al wrap 0.051 cm

$$\mu_{\text{air}} = \frac{10.16}{3.67} \text{ g/cm}^3$$

Mass attenuation coefficients from 1970 Rad Health Handbook

TABLE 2		
Energy, keV	$(\mu/\rho)_{\text{air}}$ , cm <sup>2</sup> /g	P
15	47.4	1.00
20	22.3	1.00
30	7.45	1.00
40	16.3	1.00
50	10.7	1.00
60	6.82	1.00
80	3.12	1.00
100	1.72	1.00
150	0.625	1.00
200	0.334	1.00
300	0.167	1.00
400	0.117	0.99
500	0.0955	0.97
600	0.0826	0.95
800	0.0676	0.92
1,000	0.0580	0.89
1,500	0.0469	0.83
2,000	0.0413	0.79
3,000	0.0366	0.64

Relative Detector Response (RDR) = relative fluence-to-exposure rate (FRER) times probability (P) of interaction

Relative Detector Response = Fluence Rate in Probabilities of Interaction

TABLE 3			
Energy, keV	Fluence Rate	P	RDR
15	0.0517	1.00	0.0517
20	0.0969	1.00	0.0969
30	0.2288	1.00	0.2288
40	0.3908	1.00	0.3908
50	0.5208	1.00	0.5208
60	0.5708	1.00	0.5708
80	0.5297	1.00	0.5297
100	0.4329	1.00	0.4329
150	0.2658	1.00	0.2658
200	0.1868	1.00	0.1868
300	0.1157	1.00	0.1155
400	0.0845	0.99	0.0834
500	0.0673	0.97	0.0654
600	0.0563	0.95	0.0537
800	0.0433	0.92	0.0398
1,000	0.0357	0.89	0.0317
1,500	0.0261	0.83	0.0216
2,000	0.0214	0.79	0.0168
3,000	0.0163	0.74	0.0121

Estimated RSI-700 4-Liter NaI total gamma response @ 662keV 42483 cpm/μR/hr

Use same methodology and interpolating for Cs-137 response have:

Energy, keV	$(u_{\text{air}}/\rho)_{\text{air}}$ , cm <sup>2</sup> /g	FRER ~	0.0514
662	0.0294		
Energy, keV	$(\mu/\rho)_{\text{air}}$ , cm <sup>2</sup> /g	Probability =	0.95
662	0.0780		
		RDR =	0.0466

For this detector the response to another energy is based on the ratio of the relative detector response, RDR, to the Cs-137 energy

$$\text{Response} = \frac{\text{RDR}_{\text{Cs-137}}}{\text{RDR}_{\text{Energy}}} \times \text{Response}_{\text{Cs-137}}$$

TABLE 4		
Energy, keV	RDR <sub>662</sub>	RSI NaI cpm per μR/hr, E <sub>i</sub>
15	0.0517	45202
20	0.0969	84753
30	0.2288	198333
40	0.3908	341660
50	0.5208	455547
60	0.5708	499230
80	0.5297	463298
100	0.4329	378637
150	0.2658	232311
200	0.1868	163180
300	0.1155	101033
400	0.0834	72931
500	0.0654	57226
600	0.0537	46985
800	0.0398	34789
1,000	0.0317	27724
1,500	0.0216	18888
2,000	0.0168	14682
3,000	0.0121	10589

File: T188D-20150313

Assumptions

Detector is 4 in (10.16 cm) X 4 in X 16 in  
15.24 cm detector height  
15 x 28 source at 1.5 g/cm<sup>3</sup> (dry soil) - Buildup reference  
No soil cover

0.318 carbon casing @ 2.27 g/cm<sup>3</sup>  
0.051 cm aluminum wrap @ 2.7 g/cm<sup>3</sup>

Average background count rate observed to be 3,767 cps (226,018 cpm) in 5.3 uR/hr field (ROI 10, Gross Counts)  
42,483 cpm/uR/hr

Centerpoint of detector has no offset from center of source

MDCR<sub>surveyor</sub> for Cs-137 energy

42483 cpm/μR/hr  
Bkgd = 226018 cpm

Observation interval, i = 2 second  
b<sub>i</sub> = 7533.9 counts  
d' = 3.28 from Table 6.1 of NUREG-1507  
MDCR = 8540.9 cpm  
MDCR<sub>surveyor</sub> = 9003 cpm

minimum detectable exposure rate, Cs-137 =

0.21 μR/hr

$$S_i = d' \sqrt{b_i}$$

$$MDCR = S_i (60 / i)$$

$$MDCR_{SURVEYOR} = \frac{MDCR}{\sqrt{P}}$$

P=0.90

Energy, keV	MicroShield Exposure Rate, μR/hr (with buildup)	cpm/μR/hr	cpm/μR/hr (weights d)	Percent of Nat detector response
15	2.00E-04	45202	13	0.0%
20	0.00E+00	84753	0	0.0%
30	0.00E+00	198333	0	0.0%
40	5.407E-05	341660	26	0.1%
50	0.00E+00	455547	0	0.0%
60	7.964E-05	499230	56	0.1%
80	8.105E-03	463268	5285	13.1%
100	1.877E-03	378637	1000	2.5%
150	1.917E-03	232311	627	1.6%
200	3.452E-02	163190	7929	19.6%
300	2.549E-02	101033	3625	9.0%
400	3.079E-03	72931	316	0.8%
500	2.233E-02	57226	1799	4.5%
600	6.041E-02	46985	3995	9.9%
800	7.781E-02	34789	3810	9.4%
1000	1.725E-01	27724	6732	16.7%
1500	5.491E-02	18888	1460	3.6%
2000	1.546E-03	14882	32	0.1%
3000	2.458E-01	10589	3661	9.1%
Total	7.104E-01	3246968	40366	100.0%

mR/hr, from MS	
15	2.01E-07
20	
30	
40	5.407E-08
50	
60	7.96E-08
80	8.11E-06
100	1.88E-06
150	1.92E-06
200	3.45E-05
300	2.55E-05
400	3.08E-06
500	2.23E-05
600	6.04E-05
800	7.78E-05
1000	1.73E-04
1500	5.49E-05
2000	1.55E-06
3000	2.46E-04
7.104E-04	

Microshield Output				
0.015	1.79E+03	2.19E-06	2.34E-06	1.80E-07
0.04	2.24E+01	5.87E-06	1.22E-05	2.60E-08
0.06	1.51E+01	1.19E-05	4.01E-05	2.36E-08
0.08	9.44E+02	1.30E-03	5.12E-03	2.05E-06
0.1	1.55E+02	3.05E-04	1.23E-03	4.66E-07
0.15	9.19E+01	3.22E-04	1.16E-03	5.30E-07
0.2	1.19E+03	6.13E-03	1.96E-02	1.08E-05
0.3	5.70E+02	5.06E-03	1.34E-02	9.60E-06
0.4	5.18E+01	6.75E-04	1.58E-03	1.32E-06
0.5	3.03E+02	5.33E-03	1.14E-02	1.05E-05
0.6	6.95E+02	1.56E-02	3.10E+00	3.05E-05
0.8	6.97E+02	2.29E-02	4.09E-02	4.36E-05
1	1.27E+03	5.32E-02	9.36E-02	1.04E-04
1.5	2.93E+02	2.21E-02	3.26E-02	3.71E-05
2	6.64E+00	7.21E-04	1.00E-03	1.12E-06
3	7.64E+02	1.41E-01	1.81E-01	1.91E-04

Minimum Detectable Exposure Rate =

MDCR<sub>surveyor</sub> (cpm/μR/hr)  
0.223 μR/hr

Exposure Rate, MDCR<sub>surveyor</sub> =  $\frac{MDCR}{\text{Exposure Rate, MDCR}_{\text{surveyor}}}$

and MDC for Ra-226 based on a normalized 1 pCi/g in soil with density of 1.6

Scan MDC =  $\left( \frac{MDCR}{g} \right) \times \text{Normalized Count} = \frac{\text{Exposure Rate, MDCR}}{\text{Exposure Rate, MDCR}_{\text{surveyor}}}$

Scan MDC =  $\frac{0.31}{1.19E+01}$  pCi/g  
Bo/kg